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AFAL-TR-76-66

AD B017604

LASER PUMP LAMP

ILC TECHNOLOGY, INC.
164 COMMERCIAL STREET
SUNNYVALE, CA 94086

FEBRUARY 1977

TECHNICAL REPORT AFAL-TR-76-66
FINAL REPORT FOR THE PERIOD DECEMBER 1974 - OCTOBER 1975

AIR FORCE AVIONICS LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
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This technical report has been reviewed and is approved for publication.

James M. Heitman

JAMES M. HEITMAN, Project Engr
Electro-Optic Detectors Group
Electro-Optics Technology Branch
Cloudcroft, New Mexico Facility

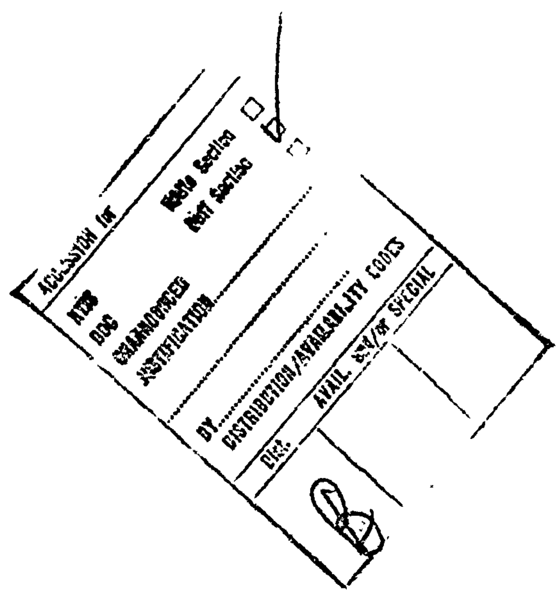
Donald J. Peacock

DONALD J. PEACOCK, Actg. Chief
Electro-Optic Detectors Group
Electro-Optics Technology Branch
Wright-Patterson AFB, OH

FOR THE COMMANDER

William C. Schoonover

WILLIAM C. SCHOONOVER, Chief
Electro-Optics Technology Branch
Electronic Technology Division
Wright-Patterson AFB, OH



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19 REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER	18 AFAL TR-76-66
2. GOVT ACCESSION NO.	
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle)	6 LASER PUMP LAMP
5. TYPE OF REPORT & PERIOD COVERED	9 Final Technical Report, Dec 74-Oct 75
6. PERFORMING ORG. REPORT NUMBER	14 R-ILC-75-2/F
7. AUTHOR(s)	10 Norman C. Anderson
8. CONTRACT OR GRANT NUMBER(s)	15 F33615-75-C-1080 NEW
9. PERFORMING ORGANIZATION NAME AND ADDRESS	ILC Technology, Inc. 164 Commercial Street Sunnyvale, California 94086
10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS	202801
11. CONTROLLING OFFICE NAME AND ADDRESS	Air Force Avionics Laboratory AFAL/DHO Wright Patterson AFB, OH 45433
12. REPORT DATE	11 February 1977
13. NUMBER OF PAGES	61
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	12 60p.
15. SECURITY CLASS (of this report)	Unclassified
15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	

16. DISTRIBUTION STATEMENT (of this Report)

Distribution limited to U.S. Government Agencies only, Test and Evaluation, July, 1975. Other requests for this document may be referred to AFAL/DHO, Wright Patterson AFB, OH 45433.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

16 2028 17 01

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18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Lasers
Optical Pumping
Arc Lamps
Alkali Vapor Lamps
Space Communications

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A program was conducted to continue development of a potassium-rubidium arc lamp for laser pumping. The average useful lifetime of such lamps was significantly increased, primarily as the result of improved lamp end seal technology. Lifetimes of nickel and Kovar endcap lamps built at the end of the program were typically in the 600-1000 hour range. The primary lamp failure mode was basal plane cleavage cracking of the sapphire envelopes.
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Difficulty in obtaining good CVD niobium-to-sapphire seal assemblies from outside suppliers hampered development work on the alternative protected end seal lamp design. One lamp was life tested, failing at 182 hours.

Experiments on laser pumping efficiency were conducted. Efficiency was found to be roughly proportional to the potassium:rubidium ratio in the lamp fill. Four millimeter bore lamps were found to be more efficient than standard five millimeter bore lamps.

FOREWORD

The work reported herein was performed under Contract F33615-75-C-1080, Project 202801 for the Air Force Avionics Laboratory, 405B Group, Wright Patterson AFB, Ohio. The Air Force technical monitor was Mr. James Heitman (AFAL/405B).

The program was carried out from December 1974 to October 1975 in the Engineering Division of ILC Technology, Inc., Sunnyvale, California 94086, which is directed by Dr. Leonard Reed.

Mr. Norman Anderson was principal investigator. Ms. Linda McLaughlin was responsible for lamp fabrication and testing, with assistance from Mr. William Warren, Ms. Pamela Pelikan, and Mr. Joseph Robillard. Mr. Heitman of the Air Force conducted experiments on lamp efficiency and also provided some of the life test data included in this report. His contributions are gratefully acknowledged. The author also is indebted to Dr. David Priest for his help in the preparation of this report.

This report was submitted to the Air Force for approval on 9 January 1976.

SUMMARY

The average useful lifetime of K-Rb lamps was extended significantly on the program. In final cycled life tests on "latest technology" lamps, the average life of nickel endcap lamps was 867 hours and 86 on-off cycles. Kovar endcap lamps averaged 630 hours and 67 on-off cycles. These values compare favorably with an interim goal for the present contract period of 511 hours and 73 on-off cycles. Advances in lamp seal technology were particularly responsible for the improved lifetimes.

A principal failure mode of "latest technology" lamps was basal plane cleavage cracking in the sapphire envelope. This cracking is possibly induced by chemical attack of the sapphire by potassium, in particular the growth of potassium beta alumina into the sapphire lattice along basal planes. Envelope frosting was also observed, especially in certain lamps tested in vacuum to simulate the absence of free convection cooling in satellites. A direct reaction between potassium vapor and the sapphire to form potassium beta alumina seems plausible. The cleavage cracking and frosting phenomena must be given considerable attention in future lamp development work.

The protected end seal (PES) lamp was redesigned to improve fabrication ability and incorporate CVD niobium endcap seals and calcium aluminate frit seals. Manufacturing problems encountered by both suppliers of CVD seals hampered development of the lamp at ILC. Nevertheless, a prototype lamp was successfully fabricated and life tested (surviving for 182 hours).

Comparative laser pumping efficiency measurements with 4 mm and 5 mm bore K-Rb lamps showed the former to be more efficient, as expected. In related work, it was found that lamp efficiency steadily improved with an increasing ratio of potassium to rubidium in the fill.

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I. INTRODUCTION

This final technical report provides an account of work accomplished on the most recent potassium-rubidium (K-Rb) lamp development program at ILC Technology, Inc. The program was part of the EFM (Engineering Feasibility Model) phase of the Air Force Advanced Development Program (ADP/405B). The overall goal of the Air Force effort is to establish a high data rate intersatellite laser communications system using both sun-pumped and lamp-pumped Nd:YAG lasers. The lamp-pumped laser requires a 250 watt CW pump lamp with a lifetime of 3000 hours. K-Rb arc lamps are presently the most efficient and are potentially the most long-lived lamps for this application.

The primary objective of this program was to increase the useful lifetime of such lamps. An interim goal of 511 hours mean time before failure and 73 on-off cycles was established for the present contract.

In parallel with the subject program, ILC also conducted related lamp development work under subcontracts with McDonnell Douglas Astronautics Company and GTE Sylvania. Because of the considerable interplay among the three programs at ILC, this report, for the sake of completeness, draws also from the McDonnell Douglas and Sylvania sponsored efforts.

A brief review of earlier work is given in Section II of this report. Development work on protected end seal (PES), nickel endcap, and Kovar endcap lamp types is described in Sections III, IV, and V, respectively. In Section VI, various general lamp topics are discussed, including degradation of the lamp envelopes and results of recent experiments on laser pumping efficiency.

II. REVIEW OF PREVIOUS WORK

Early work at ILC Technology on K-Rb lamps [1-3] was primarily devoted to studies of the effects of design and operational variables on laser pumping efficiency. Lamps at that time employed translucent alumina, drawn tubular sapphire, or cored and polished sapphire envelopes with brazed niobium endcaps. The lamps were operated in evacuated bell jars, in pump cavities purged with inert gas, or in sealed off fused quartz vacuum jackets to prevent oxidation of the endcaps.

Because of the difficulty of maintaining a sufficiently pure gas cover in the purged cavity approach and because of the excessive size and possibly inferior laser pumping efficiency of double envelope lamps, a single envelope air compatible lamp, the protected end seal (PES) lamp was later developed. In the PES design, hermetic secondary ceramic-metal end enclosures are frit-sealed to the primary sapphire envelope and evacuated to protect the niobium endcaps. Early PES lamps were short-lived, primarily because of unreliability of the solder glass frit seals then used. They were also subject to the general problem of "frosting" of the envelope interior surface caused by chemical attack of the sapphire by K-Rb fill.

On the first EFM phase K-Rb lamp development program at ILC,[4] frosting of envelopes early in life was essentially eliminated by improved lamp bakeout and filling procedures and by the use of an internal uranium getter. Difficulty was encountered in fabricating a redesigned (EFM laser-compatible) PES lamp due to cracking of the solder glass frit seal during final bakeout. New, high temperature calcium aluminate frit seals were evaluated as replacements for the solder glass seal. Early findings were encouraging. A new PES lamp endcap seal concept involving deposition of the niobium endcaps directly onto the sapphire by chemical vapor deposition (CVD) was also explored with favorable results.

Problems with the PES lamp prompted the introduction of an alternative lamp design, the nickel endcap lamp, during the program. This lamp has a simple, bare endcap configuration and is able to operate in air because of the intrinsic oxidation resistance of the nickel end members and the ILC-proprietary brazed seals employed for endcap attachment.

All lamps on the program were designed specifically for operation with the EFM laser. All had cored, polished sapphire envelopes for best laser pumping efficiency and were equipped with integral auxiliary heaters

to facilitate control of the K-Rb reservoir temperature. At the end of the program, the average lifetime of nickel endcap lamps was 35 operating cycles and over 200 hours (with substantial statistical scatter).

Despite the considerable progress made on the last program, lamp lifetime was still far from adequate; more work was required to extend multi-cycle lifetime into the 1000 hour regime. A continuation of iterative development work on the nickel endcap lamp and incorporation of new seal technology on the PES lamp were planned as major parts of the present program.

III. DEVELOPMENT OF PROTECTED END SEAL LAMPS

A. General Remarks

As noted earlier, problems with the solder glass frit seal incumbered fabrication of PES lamps on the previous program. Accordingly, the use of a stronger, more refractory calcium aluminate glass frit seal was a major design requirement on this program. In addition, a decision was made to employ CVD niobium endcap seal on the lamps, largely because the brazed seal previously used has a melting point too low to withstand the new high temperature frit sealing operation. The lamp configuration was modified to accommodate the new sealing technology and to make fabrication easier. The lamp design is described in Section III-B.

Before attempting to build lamps of the modified design, testing of high temperature glass frit seals and CVD seals was conducted using sample geometries representative of the seal configurations in the lamp. Results of this work, discussed in Section III-C, were generally favorable.

Problems developed when fabrication of lamps was undertaken. In particular, suppliers of CVD seals encountered difficulties in producing acceptable parts for lamp use. Most of the early units had cracks in the sapphire beneath the seal or were not vacuum tight. The delays associated with reworking or replacing these parts limited the number of lamp fabrication attempts at ILC. Lamp fabrication work and the problems encountered in this effort are discussed in Section III-D.

One lamp was successfully fabricated and put on life test. It operated for 182 hours before a leak developed in a copper-brazed end bell joint. This result is encouraging considering that the lamp was the first of its kind to be tested.

Despite the difficulty in successfully fabricating PES lamps, the design concept remains attractive because the PES lamp in principle is more resistant than nickel and Kovar endcap lamps to long term deterioration at elevated temperatures. Aside from problems of fabrication yield, the CVD niobium endcap seals and calcium aluminate frit seals appear to function quite satisfactorily under real or simulated lamp service conditions. The end bell seals, which operate at lower temperatures than the critical endcap and frit seals, can be made quite reliable using conventional technology.

It is recommended that development work on the PES lamp be continued. Straightforward modifications to frit seal and end bell seal designs and nominal development work on CVD seal fabrication are likely to improve lamp yields and lifetimes substantially.

B. Design

The redesigned PES lamp is shown in Figure 1. The design employs both high temperature glass frit seals and CVD endcap seals. The envelope configuration reflects a desire to use butt-type frit seal joints, rather than lap-type joints used on previous designs. To provide room for a shoulder on the sapphire envelope for the butt joint, the envelope is flared out at each end.

The lamp fabrication sequence, illustrated in Figure 2, is as follows:

1. The sapphire envelope is procured in a preliminary configuration (Figure 2A).
2. Niobium sleeves are deposited onto each end of the envelope by chemical vapor deposition (Figure 2B).
3. The outside of the envelope is then ground and polished to final shape (Figure 2C). This operation is performed after the CVD step to allow removal of chemically etched areas, resulting from the CVD process, on the exterior of the sapphire.
4. Electrode subassemblies are attached to the envelope assembly by TIG welding in an argon atmosphere glove box.
5. Sapphire end bell sleeves are then frit-sealed to the lamp (Figure 2D) at high temperature in vacuum.
6. Nickel end bell members are brazed to the premetallized sapphire sleeves with pure copper (Figure 2E).
7. The lamp is then baked out in vacuum at 600°C, filled with K-Rb and xenon and pinched off.
8. The heater mount is TIG welded to the cathode end of the lamp.
9. The end bells are evacuated (with an argon backfill optional) and pinched off (Figure 2F).

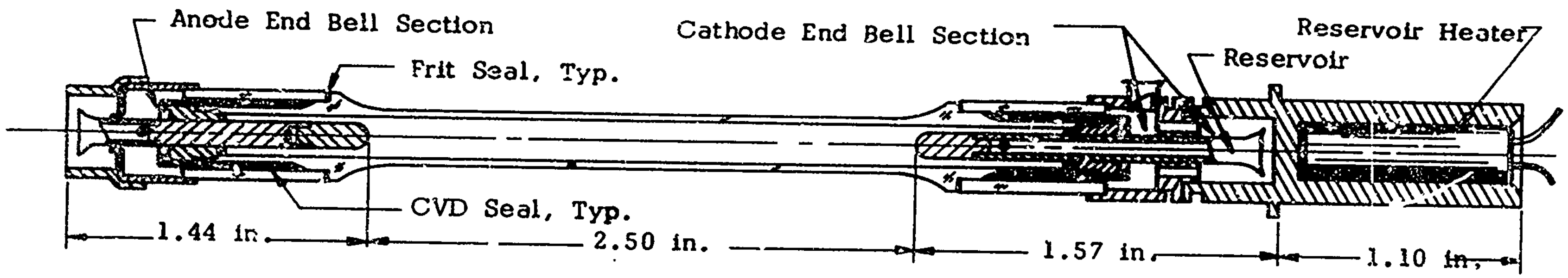

FIGURE 1. PES Lamp Design

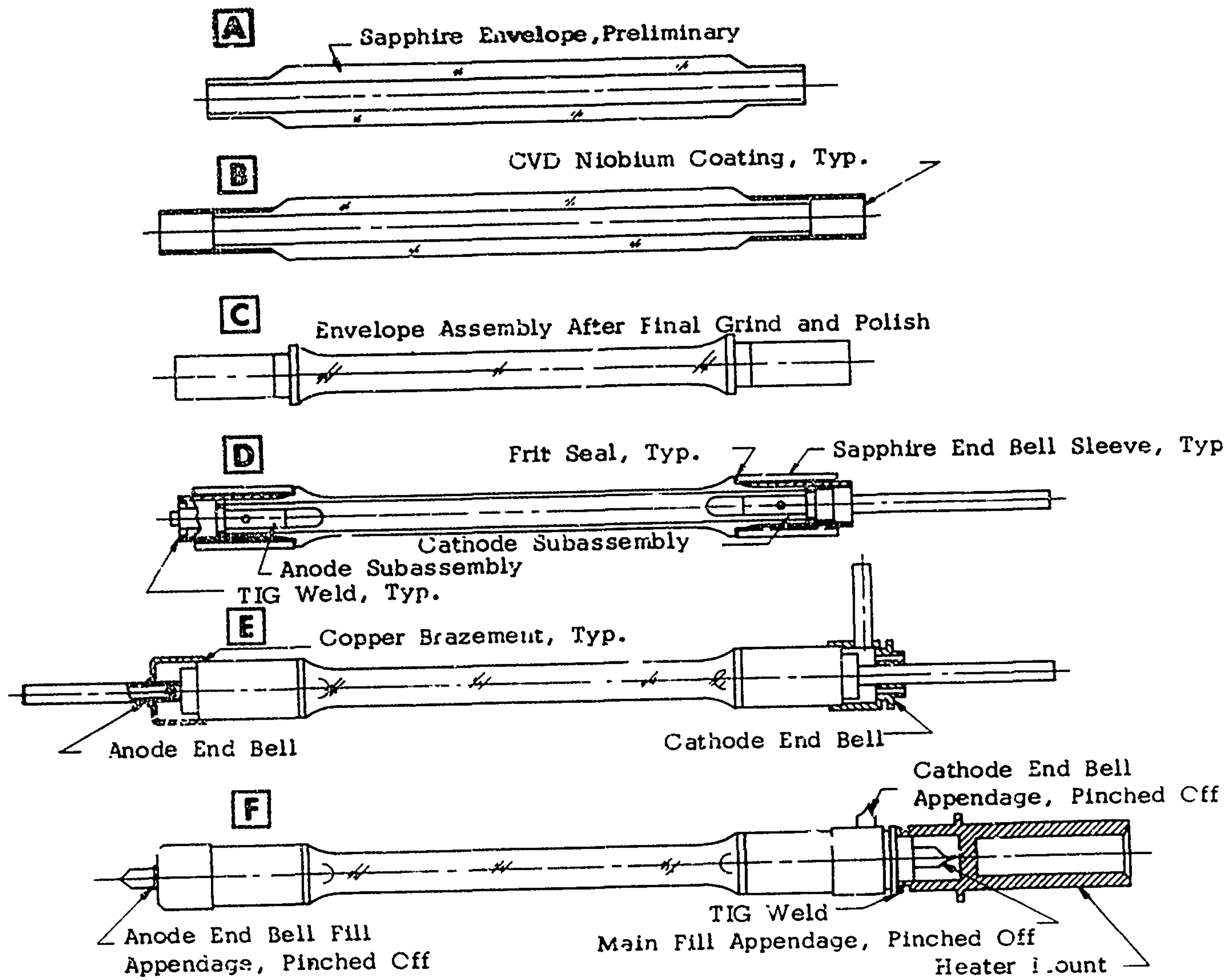

FIGURE 2. PES Lamp Fabrication Sequence

C. Design Support

1. Frit Seals

Four candidate glasses for use in PES lamp frit seals were already under investigation at the beginning of the program. These were:

1. Corning Code 1731 - a commercially available sealing glass developed for use in Nb-to-Al_2O_3 and Ta-to-Al_2O_3 seals; 1590°C sealing temperature.

2. Kingman Feldspar - a relatively pure mineral of the composition $K_2O \cdot Al_2O_3 \cdot 6SiO_2$ that had been used successfully elsewhere for joining sapphire parts[5]; 1425°C nominal sealing temperature.

3. Calcium-alumino-silicate (CAS) - a composition developed by Klomp and Botden[6] for use in alumina seals with the composition (by weight percent) $42CaO$-$18Al_2O_3$-$40SiO_2$, synthesized at ILC; 1425°C nominal sealing temperature.

4. PF-3 glass - a proprietary calcium aluminate sealing glass provided to ILC by the GTE Sylvania Lighting Division, used for Nb-to-Al_2O_3 seals in sodium street lamps; 1570°C sealing temperature.

All of these glasses have softening temperatures much higher than the service temperature of frit seal joints in PES lamps. The thermal cycle durability of seals made with the above glasses would be the primary measure of their relative suitabilities for lamp use. In addition, their amenability to sealing under vacuum was of strong interest because of the much reduced likelihood of interstitial (oxygen, nitrogen) pick-up and embrittlement of the niobium sleeves on the lamps when heated in vacuum rather than under an "inert" cover atmosphere.

Test seal assemblies consisting of sapphire rings butted together were fabricated. Seals made with Corning Code 1731 glass exhibited delayed cracking; i.e., often during cooldown. Evaluation of this glass was, therefore, discontinued. Feldspar seals were also dropped from consideration when it was found that the molten glass severely frothed in vacuum, probably because of the high vapor pressure of the K_2O constituent.

Good early results were obtained with the CAS and PF-3 sealing glasses. Seals with both glasses could be made under vacuum although the CAS glass had to be premelted onto one of the mating seal surfaces under atmospheric pressure to prevent bubble formation in the subsequent vacuum sealing operation.

Test frit seal assemblies made with CAS and PF-3 sealing glasses were subjected to repetitive thermal cycling between room temperature and 700°C (the anticipated lamp service temperature). In these tests, conducted in vertical clam shell furnaces, a motor-driven arrangement was used to automatically raise and lower the specimens into and out of the furnace in twenty-minute cycles. PF-3 seals survived several hundred such cycles with no apparent degradation. By contrast, one of two CAS seals developed cracks after 350 cycles. Based on these results, the PF-3 glass was selected for further testing.

In the second phase of testing, seal specimens having a joint geometry identical to that of the frit seals in lamps were fabricated. The specimen geometry is shown in Figure 3. Four sealed assemblies of this design were made with the PF-3 glass. Two had cracks in the sapphire and glass after sealing. The cracking was attributed to an overly tight fit between the specimens and the improvised fixture used to hold the parts during the sealing operation. The other two seals were free of defects and remained vacuum tight after over 1900 thermal cycles.

Based on these results, PF-3 glass frit seals of the original design configuration were used in fabricated lamps.

2. <u>CVD Seals</u>

Initial evaluation of niobium-to-sapphire seals made by chemical vapor deposition (CVD) was undertaken on the previous program. The seal was of interest primarily because of its ability to withstand the subsequent high temperature frit sealing operation.

The CVD seal was first tested on this program in the form of short trial assemblies having a representative joint geometry (Figure 4). CVD sealed trial assemblies were procured from two suppliers. As-received trial assemblies were helium leak-checked and carefully inspected visually for defects. Selected trial assemblies were heated in vacuum to 1500°C and again in vacuum to 1100°C to simulate the frit sealing and copper brazing operations that the CVD seals must endure during lamp fabrication. They were then enclosed under vacuum in fused quartz capsules and subjected to the thermal cycle testing described above.

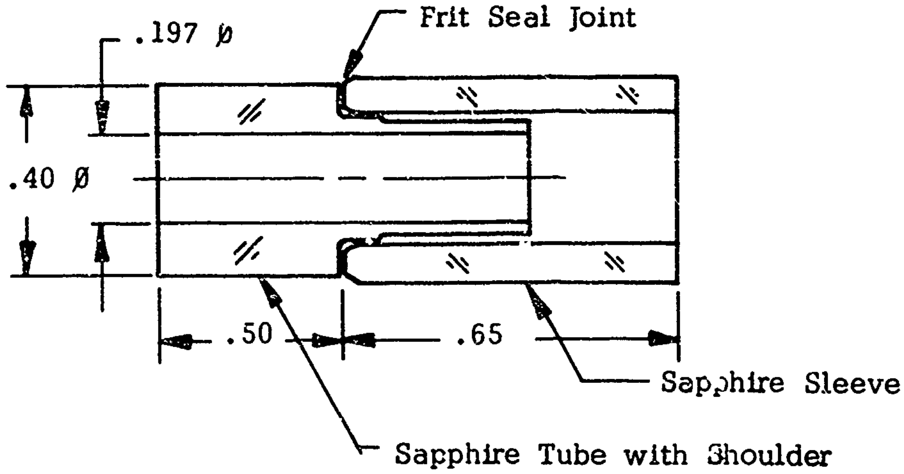

FIGURE 3. Frit Seal Test Specimen

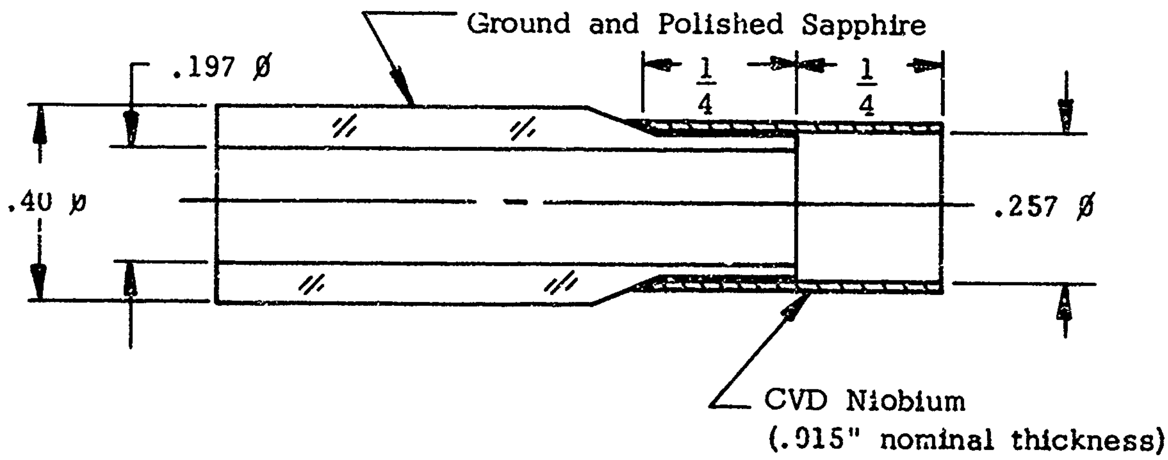

FIGURE 4. CVD Seal Test Specimen

Results of the overall evaluation are summarized in Table 1. Seals from both suppliers survived the thermal cycle sequence described above without apparent degradation.

Because cracks were detected in the sapphire under the seal area in two specimens obtained from one supplier, the other supplier was selected to provide full length "double ended" envelope assemblies for lamp fabrication.

TABLE 1

RESULTS OF CVD SEAL EVALUATION

	Supplier A	Supplier B
No. of samples	5	2
Leak check	4 of 5 vacuum tight	2 of 2 vacuum tight
Weld test	1 attempted and successful	1 attempted and successful
$1500^{\circ}C$ thermal cycle	1 sample cycled twice; still vacuum tight	1 sample cycled twice; still vacuum tight
$1100^{\circ}C$ thermal cycle	above sample cycled once; still vacuum tight	above sample cycled once; still vacuum tight
$700^{\circ}C$ thermal cycle	above sample cycled 423 times; still vacuum tight	above sample cycled 423 times; still vacuum tight
Visual inspection	minimal etching of sapphire; no apparent cracks	considerable etching; one sample has cracks in sapphire

D. Fabrication

Five lamp fabrication starts were planned in the first prototype lot. Five CVD-sealed lamp envelope assemblies were ordered from the previously selected supplier. Meanwhile, other piece parts were procured or machined for these starts.

When the envelope assemblies were received, only one was found to be acceptable. In the other assemblies, at least one of the CVD seals was nonhermetic or the deposited niobium was brittle (as determined

by a "squash test" performed on short rings from each deposited niobium sleeve). The unacceptable assemblies were returned to the supplier for rework.

The supplier attempted to recover the sapphire tubes from the rejected envelope assemblies for reuse by dissolving the deposited niobium in an HF solution. Removal of the niobium revealed the presence of cracks in the underlying sapphire. The supplier attributed these cracks to stresses caused by reheating of the assemblies during deposition of the second sleeve in their two step (one end at a time) process. It was suggested that by chemically dissolving the molybdenum mandrel under the extended section of the first sleeve before depositing the second sleeve, these stresses could be eliminated.

Supplementary funding was obtained through the McDonnell Douglas subcontract to allow procurement of five additional envelope assemblies from each of the original suppliers. The procurements provided for preliminary engineering work by both suppliers prior to fabrication of the new envelope assemblies.

Results of the second procurement were still somewhat disappointing, but reflected finite improvement in the quality of delivered units. One supplier delivered three acceptable envelope assemblies, lost two in process, and took two back for rework due to helium leaks in the seals. One of the reworked assemblies was subsequently delivered in acceptable condition; the other still leaked.

The other supplier delivered three assemblies. Two had helium leaks and were returned for rework. The other had embrittled niobium at one end. After an 1100°C vacuum firing at ILC, the niobium became quite ductile indicating that dissolved hydrogen (picked up during the CVD operation) had been removed. The effectiveness of the vacuum firing step suggests that this straightforward technique can be routinely performed on CVD niobium assemblies to ensure that the deposited niobium is ductile. One of the two reworked assemblies was later delivered in vacuum tight condition. The other still leaked.

Modifications to process fixturing by the CVD suppliers in the second round of fabrication had apparently reduced the frequency of sapphire cracking. A remaining concern is the high incidence of CVD seal leaks in the delivered assemblies. Careful helium leak checking of the nonhermetic seals had shown that the leak paths were along the seal interface rather than through the deposited niobium wall. This suggested that interfacial bonding between the niobium and the sapphire substrates was marginal. The suppliers believe that this problem is associated with

inadequate sapphire substrate temperature at the onset of the deposition process. Improved temperature control should alleviate the problem.

Due to the yield problems with the CVD seals, only four acceptable lamp envelope assemblies were received soon enough in the program to be advanced to subsequent steps in the lamp fabrication sequence. The first of these assemblies developed a seal leak during the electrode subassembly welding operation. During the frit sealing step that followed (performed to verify the alignment fixture design despite the leak in the CVD seal), the sapphire cracked adjacent to one seal. The second assembly was broken while a ductility test ring was being machined from one end.

Cracking of the sapphire adjacent to one frit seal joint occurred in the third assembly during the frit sealing operation. This recurrent problem seemed to be associated with flow of the molten glass from the shoulder seal surface into the small radial gap between the sapphire envelope and sleeve. Given the presumably good thermal expansion match between the sealing glass and the sapphire, the development of seal stresses sufficient to cause the sapphire to crack is not precisely understood. Nevertheless, the problem seems amenable to solution by a modification of the joint configuration to eliminate the narrow gap next to the butted sapphire surfaces and the capillary effects associated with such gaps.

One frit seal on the fourth assembly had a small gap after an initial frit sealing operation. The seal was successfully patched by addition of more frit and a second furnace heating sealing operation. Subsequent brazing and welding operations successfully produced a PES lamp (Figure 5) suitable for life testing. The lamp was filled with 50K-50Rb (by weight percent) and 760 torr xenon.

E. <u>Testing</u>

Lamp Number 501, the only PES lamp successfully fabricated on the program, was placed on cycled life test in air. After 18 on-off cycles and 182 hours of lamp operation, the sapphire end bell sleeve at the anode end cracked adjacent to the copper-brazed sapphire-to-nickel joint (Figure 6), allowing air to leak in and oxidize the primary niobium endcap. Excessive stress on this seal was probably caused by either the substantial thermal expansion mismatch between the nickel and the sapphire or by "self constraint" in the end bell structure due to the rigid, parallel mechanical attachments; i.e., along the outer wall and via the on-axis tubulation. Straightforward modifications to the lamp design can rectify the problem.

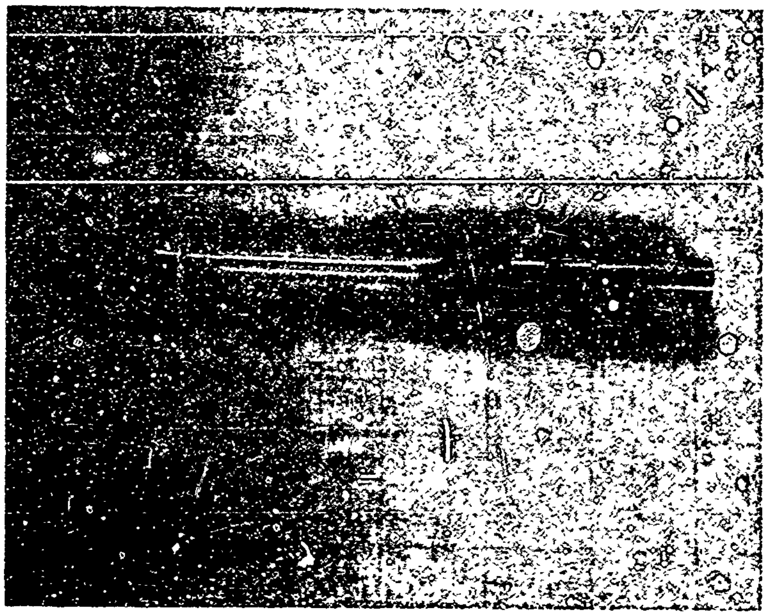

FIGURE 5. PES Lamp

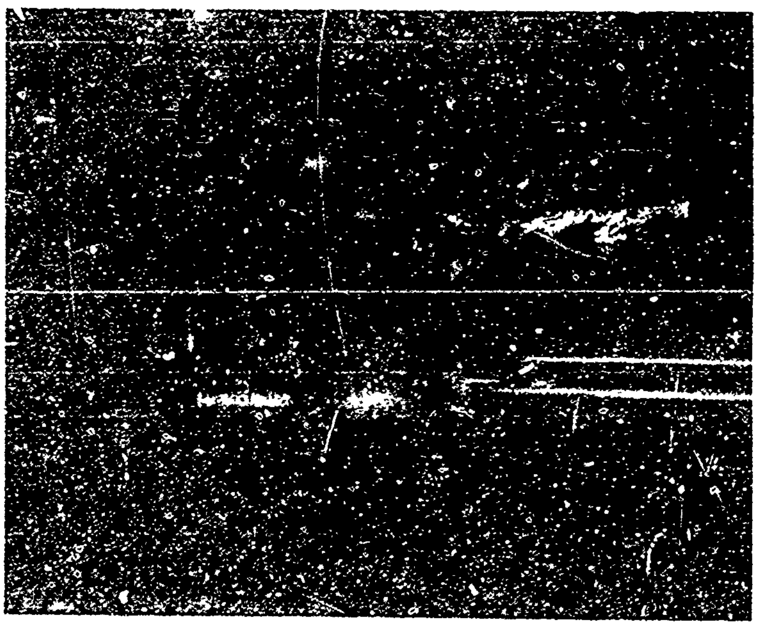

FIGURE 6. Endbell Seal Crack in PES Lamp After Testing

In general, the 182 hour lifetime for this first PES lamp put on test is encouraging. Survival of the "critical" high temperature seals; i.e., the frit seals and CVD seals, is particularly noteworthy.

IV. DEVELOPMENT OF NICKEL ENDCAP LAMPS

A. General Remarks

The first nickel endcap lamps fabricated on the previous program were extremely sensitive to on-off cycling, generally failing within a few cycles by fracture in the sapphire adjacent to the seals. The problem was traced to excessive mechanical stress resulting from the large thermal expansion mismatch between the sapphire and the relatively hard braze-affected zone in the nickel endcap. To reduce stress levels in the sapphire, tungsten stress compensating washers, approximately six mils thick, were incorporated in the nickel lamp seals. This modification resulted in significantly better average cycle lifetimes (35 cycles) for a group of lamps tested at the end of the program. However, there was wide variability in the results with one lamp surviving 157 operating cycles while others failed in as few as three cycles.

A main task on the present program was to continue development work on the nickel lamp seals to improve both average cycle lifetime and consistency. This work, described in Section IV-C, was quite successful as reflected in the markedly improved lifetimes of "latest technology" nickel endcap lamps built and tested at the end of the program.

Two fabrication lots of "latest technology" nickel endcap lamps were life tested. Mean time before failure for the first lot (of three lamps) exceeded 1000 hours with two failures occurring due to cleavage cracking in the sapphire envelope, a phenomenon presumably unrelated to the lamp end seal design. One of two lamps in the other lot developed a leak during final processing, while the other failed comparatively early in life. These results demonstrate both the long lifetime potential of nickel endcap lamps and the need to establish improved materials and process controls to avoid "bad lots" of lamps.

Other improvements to the nickel endcap lamp design were made during the course of the work, which are described in Section IV-B. Fabrication of lamps is briefly discussed in Section IV-D. Detailed results of life tests are presented in Section IV-E.

B. Design

1. Interim Lamps

The first nickel endcap lamps built on the present program, Numbers 481 and 482, were similar in design to the nickel lamps tested at

the end of the previous program including use of tungsten stress compensation washers in the endcap seals. The following modifications were incorporated in these lamps:

1. Use of brazed (with a palladium-cobalt alloy) instead of welded joints in the electrode subassemblies.

2. A 1/4 inch reduction in lamp length at the anode end for purposes of EFM laser interfacing (reflecting a late design change in the laser cavity lamp mounting provisions).

3. Use of sapphire envelopes with polished instead of ground finish end seal surfaces (prompted by favorable results with polished seal surfaces on the seal development task).

2. Latest Technology Lamps

Near the end of the program, a group of nickel endcap lamps (Numbers 491, 492, 493, 502, 503) was fabricated for final life testing. The life tests were designed to provide an assessment of the nickel lamp in its "latest technology" embodiment. These "latest technology" lamps (Figure 7) incorporated the following design modifications:

1. Use of a "low zirconium" variation of the brazed endcap seal found promising in work on seal development.

2. Use of electrode subassembly weld caps made from nickel sheet stock instead of rod stock as had been used previously. This modification was made to prevent the occurrence of "pipe" leaks common in wrought metal structures parallel to the forming direction and which had been encountered frequently in earlier subassemblies. (Use of sheet stock orients such potential leak paths crosswise in the caps and thus negates their potential effect.)

3. Use of backup rings in the endcap seals with polished seal surfaces instead of the ground finish used previously.

3. Four Millimeter Bore Lamps

Upon completion of "latest technology" lamps, several lamps with 4 mm bore diameter envelopes were fabricated. These lamps were used in laser pumping efficiency tests to compare 4 mm against the standard 5 mm bore lamps. Four millimeter bore lamps with envelopes of both

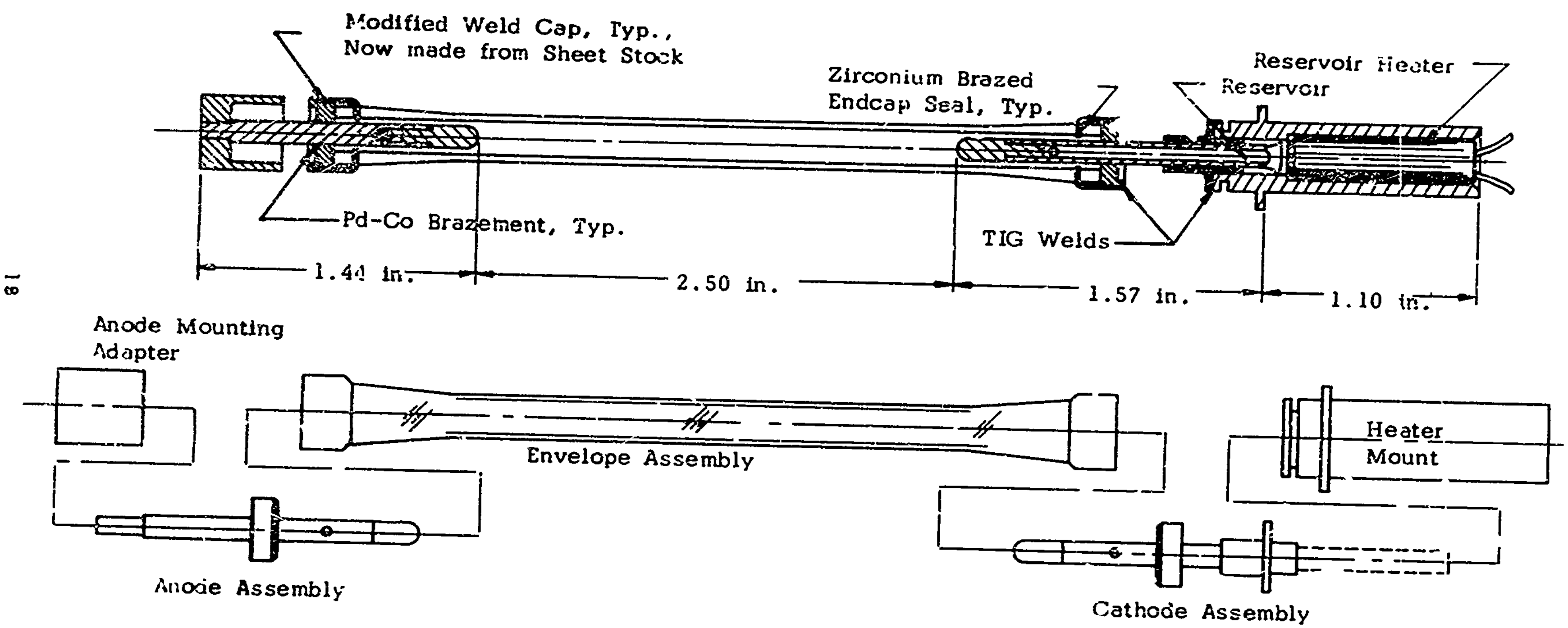

FIGURE 7. Latest Technology Nickel Endcap Lamp Design

standard 0.030 inch wall thickness (so-called 4 x 5.5 mm) and thicker 0.050 inch walls (4 x 6.5 mm) were built. It was hoped that the larger o.d. envelope with its proportionately greater external surface area for heat dissipation would offset the increase in internal envelope wall temperature expected in reducing the bore diameter from 5 to 4 mm.

The designs of the 4 mm bore lamps, aside from obvious differences in envelope dimensions, were virtually identical to that of the "latest technology" 5 mm bore lamps.

C. Design Support (Seal Development)

Although, as noted earlier, the employment of tungsten stress compensation washers in nickel lamp seals had improved the average cycle lifetime of lamps, considerable variability in the quality of the seals remained a problem. In an attempt to improve the consistency of seals, the use of sapphire with polished rather than ground finish seal surfaces was explored. It was believed that premature failures in seals were due to the presence or generation of cracks at spots on the sapphire seal surface unwetted by the brazing alloy. High localized stresses might exist in these regions owing to the abrupt discontinuities between bonded and unbonded areas. The rough, flawed topography of the ground finish surface seemed to be both more likely to inhibit complete wetting and more vulnerable to attendant cracking; i.e., notch sensitive.

Evaluation of seals with polished surfaces was carried out using short trial seal specimens (Figure 8) which were tested by thermal cycling in the standard manner (see Section III-C). An improvement in cycle lifetime over similar specimens with ground finish seal surfaces was noted as shown in Table 2. However, the inconsistency characteristic of earlier versions of the tungsten stress compensated seal remained. In addition, interfacial wetting in the seals was still incomplete.

It was decided to discontinue use of tungsten compensation washers in the seals in favor of a more straightforward approach involving reducing the amount of zirconium used in the seal. Less zirconium, it was reasoned, would result in a less extensive braze-affected zone in the nickel with consequently reduced seal stress. However, it was expected that wetting of the sapphire seal surface, already marginal, would be even more of a problem if the amount of zirconium used in the seals was significantly reduced. Thus, the goal was to develop a "low" zirconium seal with improved sapphire wetting.

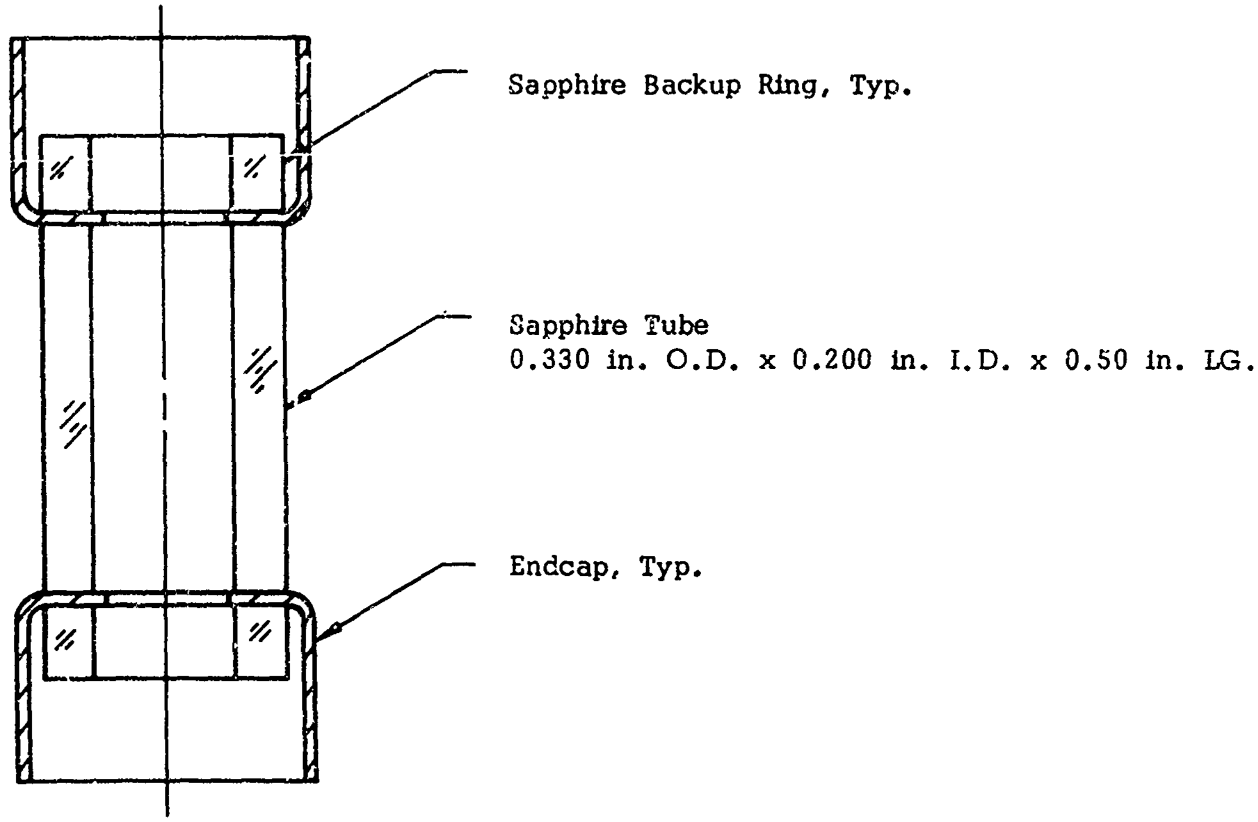

FIGURE 8. Brazed Seal Test Specimen

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TABLE 2

RESULTS OF NICKEL-TO-SAPPHIRE SEAL TRIALS

Group	Seal Surface Finish	Tungsten Compensation	No. of Samples	Avg. Cycles Before Failure	Std. Dev.
1	Ground	Yes	3	24	21
2	Polished	Yes	16	374	610
3	Polished	No (use thin Zr)	14	1025*	121*

*Based on incomplete test results; ten samples still intact after extensive testing.

A brazing parameter study was conducted that included the evaluation of the effects of critical brazing variables such as the time/temperature schedule, amount of zirconium, and vacuum level. The most important finding of the study was that if an intermediate vacuum level was maintained during brazing (obtained by throttling the ion pump valve on the vacuum brazing furnace) complete seal wetting was achieved, even with only half as much zirconium as had been originally used in seals. Thermal cycle tests on the well-wetted, low zirconium seals showed them to be extremely durable. As shown in Table 2, nearly all seals of this type survived in excess of 1000 thermal cycles. An effect of the brazing time/temperature schedule on wetting of seals was also observed, relatively short schedules being preferable.

D. Fabrication

1. Procedure

The fabrication of nickel endcap lamps is relatively simple in comparison with, for example, PES lamps. With reference to Figure 7 given earlier, the fabrication sequence is as follows:

1. Nickel cups are attached to the sapphire envelope by zirconium brazing in vacuum.

2. The electrode subassemblies are fabricated by TIG welding the tungsten electrode tips to the nickel supports (note: all lamp welds are done in a purified argon glove box) and subsequently attaching the end weld caps (plus a tubulation weld adaptor on the cathode subassembly) by brazing with a palladium-cobalt brazing alloy.

3. The electrode subassemblies are joined to the envelope assembly by TIG welding.

4. The lamp is baked out under vacuum at 500°C, filled with K-Rb and xenon, and pinched off. The pinch-off is then back-welded for added reliability.

5. The heater mount and anode mounting adapters are attached to the cathode and anode ends of the lamp, respectively, by TIG welding.

6. The lamp is given a final 300°C bake to promote gettering of residual gaseous impurities by the uranium.

2. <u>Yield</u>

The fabrication yield of nickel endcap lamps was reasonably high. Three of four fabrication starts (where a start is defined as a brazed envelope assembly) on interim lamps produced operable lamps. One start was scrapped due to a poorly wetted brazed seal. Six of nine fabrication starts produced operable "latest technology" lamps. Two envelope assemblies had poorly wetted seals after envelope brazing and were scrapped. Two others (Numbers 494 and 495) had marginally wetted seals and were used as "expendable" lamps in power supply tests and the like. One other lamp, Number 502, developed a leak during final bakeout and could not be operated. The yield of 4 mm bore nickel endcap lamps was 100 percent (seven of seven).

A summary of nickel endcap lamp fabrication is given in Table 3.

TABLE 3

SUMMARY OF NICKEL ENDCAP LAMP FABRICATION

Start No.	Serial No.	Design Type	K-Rb Ratio	Xenon Pressure (torr)	Disposition
24	480	Interim	100:0	760	Failed in first operation
25	---	Interim	75:25	760	Leak in final processing
26	481	Interim	50:50	760	Life tests at AFAL/405B
27	482	Interim	25:75	760	Life tests at ILC
39	---	Latest Tech.	-----	---	Scrapped, seals not well-wetted
40	---	Latest Tech.	-----	---	Scrapped, seals not well-wetted
41	494	Latest Tech.	75:25	760	Misc. use at ILC
42	495	Latest Tech.	75:25	760	Life test in vacuum at ILC
43	491	Latest Tech.	75:25	760	Life test at ILC
44	492	Latest Tech.	75:25	760	Life test at ILC
45	493	Latest Tech.	75:25	760	Life test at ILC
71	502	Latest Tech.	75:25	760	Leak in final processing
72	503	Latest Tech.	75:25	760	Life test in ILC
73	504	4 mm bore	75:25	760	Misc. tests at MDAC
74	505	4 mm bore	75:25	760	Laser tests
75	506	4 mm bore	75:25	760	Life test by Air Force
76	507	4 mm bore	75:25	760	Life test at ILC
77	508	4 mm bore	75:25	760	Misc. tests at MDAC
78	509	4 mm bore	75:25	760	Life test by Air Force
79	510	4 mm bore	75:25	760	Laser tests

E. Testing

1. Interim Lamps

Two interim design nickel endcap lamps, Numbers 481 and 482, were life tested, one at ILC and one at AFAL/405B. The results, shown in Table 4, were somewhat disappointing. Despite the use of envelopes with polished seal surfaces, the lamps were relatively short-lived and failed in the end seals. Use of the tungsten stress compensated seal design was discontinued after these lamps had been tested. The "low zirconium" seal was developed and employed in subsequent "latest technology" lamps.

TABLE 4

LIFE TEST RESULTS FOR INTERIM DESIGN NICKEL ENDCAP LAMPS

Lamp No.	Lifetime Cycles	Lifetime Hours	Failure Mode
481	8	47	Envelope cracks at cathode end brazed seal
482	40	467	Leak in nickel endcap near cathode end brazed seal
Average Lifetime for Group:			24 cycles/257 hours

2. Latest Technology Lamps

Four "latest technology" nickel endcap lamps were life tested, all at ILC. In all tests, the lamps were operated in ambient air in open-ended fused quartz tubes (to more closely simulate the thermal conditions of laser pump cavities than could be achieved by operation in the open). Average time per operating cycle was roughly ten hours. Results of the life tests are shown in Table 5.

Lamps from the first lot averaged in excess of 1000 hours lifetime. Lamp Number 491 survived to 1536 hours, halfway to the ultimate 3000 hour goal. Two of the first lot lamps failed by parting of the envelopes along sapphire basal planes. These cracks became discernible in each lamp envelope after 400-600 hours of testing and slowly propagated

until envelope failure occurred. The subject of sapphire cleavage cracking is discussed in more detail in Section VI-A. The long-lived lamp, Number 491, developed a leak through the nickel endcap in the braze-affected zone.

The one lamp, Number 503, from the second lot that was tested failed relatively early in life (298 hours) due to the development of a leak in the braze-affected zone of the nickel endcap at the cathode end of the lamp. Subsequent metallographic examination of the leak area indicated that the leak path was along a nickel grain boundary that had been previously penetrated along its entire length (through the 10 mil wall of the endcap) by brazing alloy melt phase. Similar excessive intergranular penetration of brazing alloy was found in metallographic sections of endcaps from lamp Number 502, the other lamp in the second lot which leaked to air before testing. This suggestion of an overbrazed condition in the second lot lamps is not confirmed by checks on the brazing run records. Time/temperature and vacuum plots for the Lot 2 brazing cycle are essentially idential to the Lot 1 plots. The same brazing material and nickel for endcaps was used in both lamp lots. Apparently some more subtle variable is at play here. In any case, the lot-to-lot variation points out the need for further seal development work to identify and control all important variables in the process and establish improved consistency.

TABLE 5
LIFE TEST RESULTS FOR LATEST
TECHNOLOGY NICKEL ENDCAP LAMPS

Lamp No.	Lot No.	Lifetime Cycles	Lifetime Hours	Failure Mode
491	1	138	1532	Leak in nickel endcap near anode end brazed seal
492	1	94	830	Envelope cracking along basal plane
493	1	74	812	Envelope cracking along basal plane
503	2	36	294	Leak in braze-affected zone of cathode endcap

Average Lifetime for Lot 1: 102 cycles/1058 hours
Average Lifetime for Overall Group: 86 cycles/867 hours

3. Four Millimeter Bore Lamps

At this writing, the 4 mm bore lamps are being used in various special tests and have not been life tested.

V. DEVELOPMENT OF KOVAR ENDCAP LAMPS

A. General Remarks

Because of problems in developing reliable nickel-to-sapphire brazed seals early in the program, there was interest in evaluating Kovar alloy* as an alternate endcap material. Kovar has a much lower average thermal expansion coefficient than does nickel between room temperature and 600°C, the temperature range through which seals are cycled in typical lamp operation. Thus, it was expected that cyclic stress levels in a Kovar-to-sapphire seal would be much lower and such seals would have considerably better thermal cycle durability. Iron, nickel and cobalt, the constituents of Kovar, have low solubilities in alkali metal liquids. For this reason, it was presumed that Kovar would have adequate chemical resistance to the K-Rb fill in the lamps.

A major question was whether the zirconium brazement used in nickel-to-sapphire lamp seals could be used effectively in Kovar seals and would be adequately oxidation resistant. The long-term oxidation resistance of Kovar itself was also of some concern. Experiments were first conducted to evaluate Kovar-to-sapphire brazed seals with respect to these questions. Test results on trial seal assemblies were very encouraging. Subsequently, prototype Kovar endcap lamps were built and tested.

The primary failure mode in prototype lamps was leakage through the endcap weldments. A side experiment was conducted that clearly indicated the problem to be intergranular oxidation in the Kovar, aggrevated by thermal expansion mismatch stresses in the dissimilar metal (Kovar-to-nickel) weldment. In subsequent interim design lamps, a matched weldment (Kovar-to-Kovar) was employed. Lifetime improved substantially with one lamp surviving over 2000 hours.

"Latest technology" Kovar endcap lamps, built and tested at the end of the program, had an average lifetime of 631 hours, significantly lower than the average for interim design lamps. Failures in these lamps resulted from either basal plane cleavage cracking in the sapphire envelopes or leaks through the Kovar endcaps near the brazed end seals (much

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* Kovar, originally the trade name for the low expansion Fe-Ni-Co alloy manufactured by Westinghouse, is used in this report in the generic sense to designate the same alloy as manufactured by several suppliers, in all cases per ASTM specification F-15.

the same as the failures in "latest technology" nickel endcap lamps). Envelope cleavage cracking, a problem common to both nickel and Kovar endcap lamps, is discussed in detail in Section VI. Leaks in the braze-affected zone of the endcaps, also a common problem, is apparently induced by a combination of excessive intergranular erosion of the Kovar base metal during brazing and subsequent exposure to air at high temperature. More development work on Kovar-to-sapphire brazed seals is required to address the base metal erosion problem. It is also suggested that the requirement of life testing in air be relaxed somewhat. This is not inconsistent with the plan to provide a nitrogen cover gas for the pump lamp in satellite service.

Given the good cycle durability of Kovar endcap lamps and the survival of one lamp for more than 2000 hours in air, despite the known progressive intergranular oxidation of the Kovar, it seems likely that in a reasonably inert environment the lamp can meet the 3000 hour/400 cycle lifetime goal.

B. Design

1. Prototype and Interim Lamps

The design of prototype and interim version Kovar endcap lamps (Figure 9) was much like that of nickel endcap lamps. Indeed, prototype Kovar lamps differed from their nickel counterparts only in the use of Kovar rather than nickel seal cups.

When endcap weld failures occurred that were found to be associated with the dissimilar metals (nickel and Kovar) in the joint, the electrode subassembly weld cap material was changed to Kovar. The resulting matched metal endcap weld joint was the only modification incorporated in interim version lamps.

2. Latest Technology Lamps

Two design modifications were incorporated in "latest technology" Kovar endcap lamps (Figure 10). One modification was the use of a nickel electroplate on the endcaps to provide protection from oxidation. The other was use of electrode subassembly weld caps made from Kovar sheet stock rather than rod stock as used on interim version lamps. This modification was intended to prevent "pipe" leaks in the caps (similar to the modification made in "latest technology" nickel endcap lamps described in the previous section).

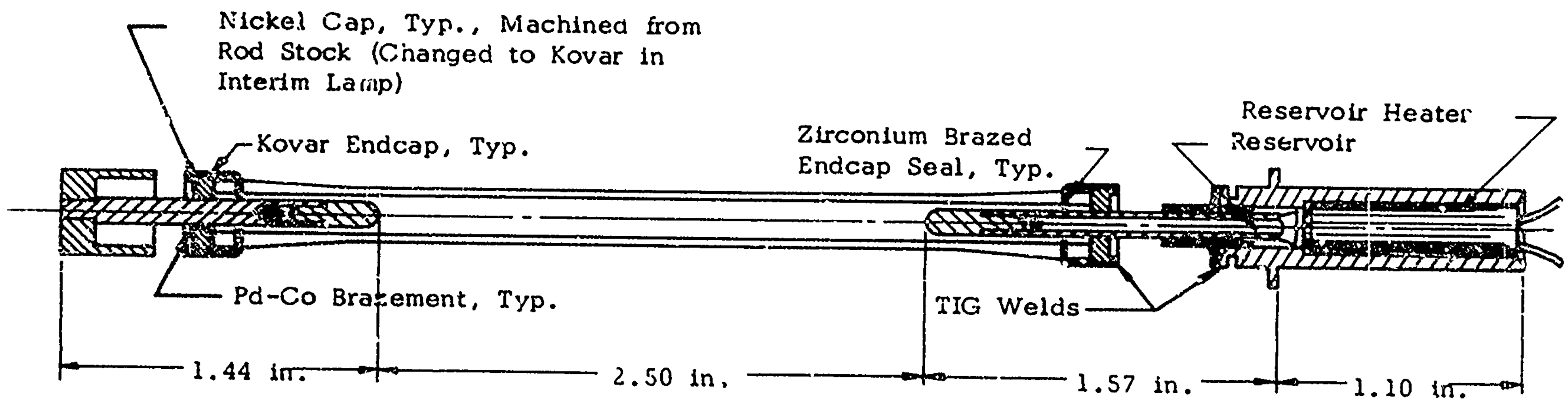

FIGURE 9. Prototype Kovar Endcap Lamp Design

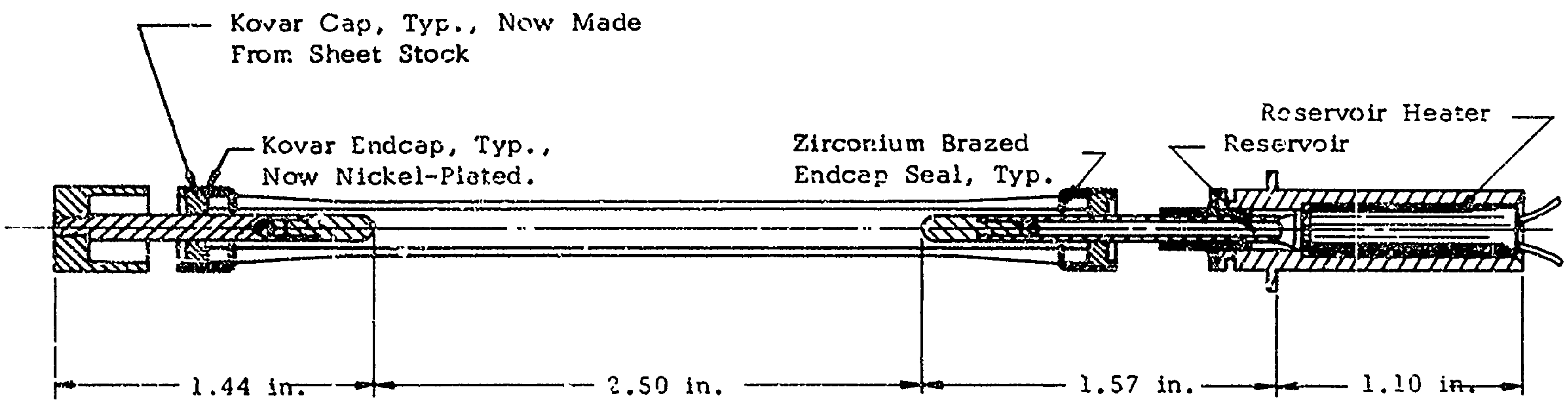

FIGURE 10. Latest Technology Kovar Endcap Lamp Design

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C. Design Support

1. Seal Development

Trial Kovar-to-sapphire seals, brazed with zirconium, were made using the standard sample configuration described in Section IV. In the first attempts, vacuum tight seals were made using varying amounts of zirconium and the same brazing schedule as used with nickel-to-sapphire seals. Many of these initial seal samples survived over 1000 thermal cycles in air.

Based on these encouraging early results, prototype Kovar endcap lamps were built and tested. Meanwhile, Kovar seal development was continued with emphasis given to rectifying apparent deficiencies in the seal: incomplete wetting of the sapphire seal surface by the brazing alloy, substantial base metal erosion by the brazing alloy, and run-out of the brazing melt on the underside of the Kovar cups.

A reduction in the amount of zirconium used in the seals seemed to be the most obvious means of minimizing base metal erosion and braze run-out. This approach was evaluated experimentally. Unfortunately, it was found that reduction in zirconium aggrevates the wetting problem. Some indications of a relationship between wetting and vacuum level during brazing were observed (as in nickel seals) but this effect could not be characterized adequately. Consequently, the brazed seals used in "latest technology" Kovar endcap lamps were little different than the original seals used in prototype lamps.

2. Corrosion and Oxidation Tests

It was hypothesized that the corrosion-like failure of endcap weldments in prototype Kovar lamps was caused by oxidation, intergranular attack by the alkali metals, or a combination of these. It was further suspected that thermal cycling may have had an influence because of thermal expansion mismatch stress in the dissimilar metal weldment area.

An experiment devised to verify these hypotheses involved the fabrication of short test capsules (Figure 11) using standard lamp parts. Both Kovar and nickel electrode subassembly caps were used to provide both matched and dissimilar metal weldments for evaluation. The capsules were processed in the same fashion as lamps and were filled with K-Rb and xenon. A uranium getter was inserted in each capsule prior to pinch-off. The capsules were then either thermal cycled between room

temperature and 650°C or held at a constant 650°C. Tests were conducted in both air and nitrogen atmospheres.

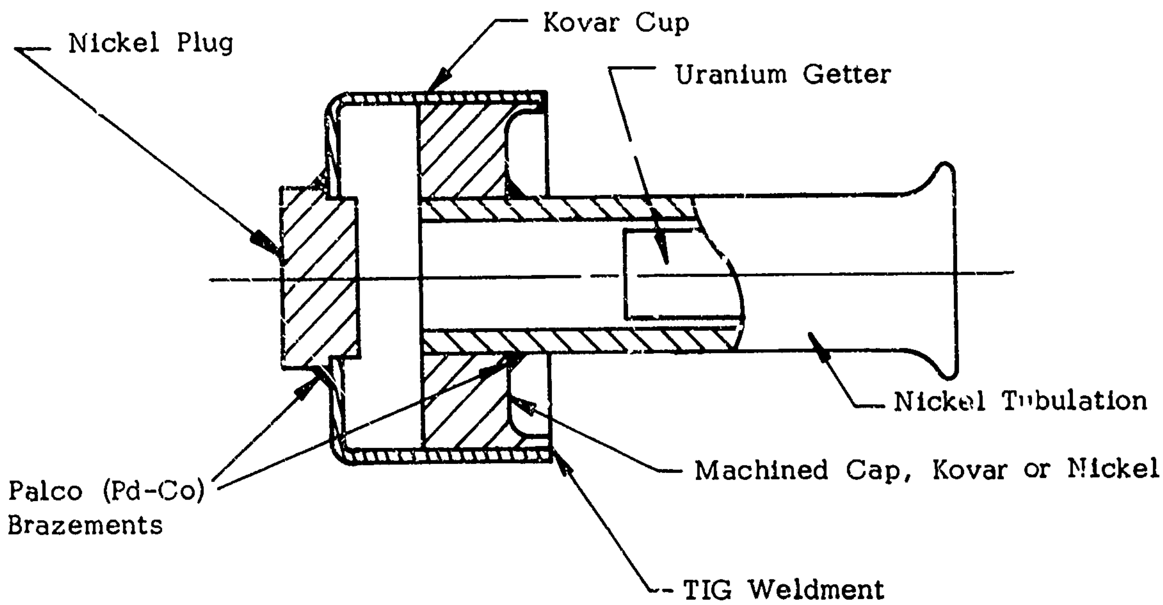
The test matrix and experimental results are shown in Table 6. The results strongly suggest that a combination of oxidation and thermal cycle stress in the weldment (due to the Kovar-nickel thermal expansion mismatch) cause failure within the weldment or in the Kovar immediately adjacent to it.

Metallography of the weldment areas amplifies this finding. As can be seen in the photomicrograph in Figure 12, extensive intergranular oxidation of the Kovar has occurred. Where tensile stress is present (i.e., adjacent to nickel members), the grain boundary oxidation is exaggerated and intergranular cracking developes (Figure 13). The occurrence of intergranular oxidation in Kovar is not surprising; a review of pertinent literature[7,8] revealed that intergranular oxidation of low expansion Fe-Ni alloys is common. A probable consequence of intergranular oxidation of the Kovar, once the oxidation has progressed to the internal surface of the Kovar member, is alkali metal attack of the oxidized grain boundaries by dissolution or redox mechanisms.

Based on the above findings, machined Kovar electrode subassembly caps were used instead of nickel caps in Kovar endcap lamps subsequently fabricated.

Work was continued on methods to reduce or eliminate the intergranular oxidation of Kovar. An experiment was conducted to evaluate nickel electroplated coatings as a means of protecting the Kovar from oxidation. A large number of Kovar endcap cups were electroplated in a chloride bath with a 0.5-1 mil layer of nickel. Half of the plated parts had been previously vacuum fired to simulate the zirconium brazing cycle. This firing operation substantially increases the grain size of the Kovar and, presumably, influences oxidation behavior as a result. Many of the plated parts were subsequently fired to various intermediate temperatures (e.g., 600°C, 800°C) in attempts to improve adhesion of the plate.

Oxidation of the plated Kovar cups was evaluated by either soaking them in air at 650°C for 300 hours or by thermally cycling them in air approximately 500 times between room temperature and 650°C. One hundred percent visual inspection and metallography on selected samples was conducted after the oxidation testing. The results were very inconclusive. Certain observations are worth noting, however:



NOTE: All specimens filled with K-Rb (50-50) and 760 torr xenon.

FIGURE 11. Kovar Corrosion Test Specimen

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TABLE 6

KOVAR CORROSION TEST SUMMARY

Sample No.	Cap Material	Test Mode*	Results
1	Kovar	steady in air	leak through "pipe" in cap after 28 hours
2	Kovar	cycled in air	leak in cup adjacent to nickel plug joint (stressed region) in 300 cycles
3	Nickel	steady in air	OK after 500 hours (no cycle stress)
4	Nickel	cycled in air	weld cracks and leakage in less than 84 cycles
5	Kovar	not tested	-----
6	Kovar	cycled in N_2	OK after 500 cycles (oxidation minimal)

*Cycle tests between room temperature and 650°C in 20 minute, full cycles; steady tests at constant 650°C.

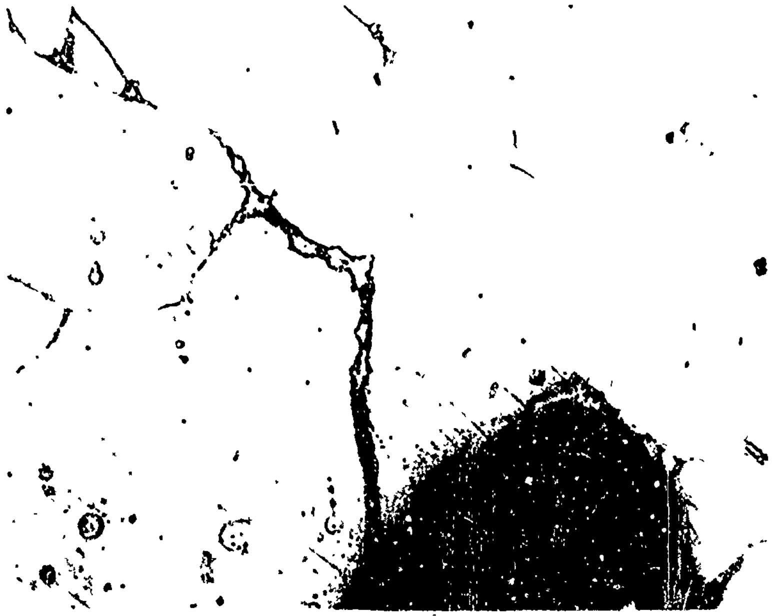

FIGURE 12. Intergranular Oxidation in Kovar (200X)

FIGURE 13. Intergranular Cracks in Kovar (200X)

1. The nickel plate was generally indiscernible in metallographic sections, particularly on specimens that had been fired after plating. High temperature interdiffusion of the nickel and underlying Kovar probably had occurred.

2. Intergranular oxidation was present on nearly every sectioned sample, an exception being one sample that had not been fired after plating.

3. There was considerable variability in the outward appearance of the samples and in the degree of oxidation as reflected in sectional views. This suggests the possibility of variability in the original plate thickness.

Despite the uncertain results derived from the above experiments, a nickel electroplate was applied to the Kovar end members on the final lamps made on the program, the so-called "latest technology" Kovar endcap lamps. It was reasoned that although the effectiveness of nickel plating had not been demonstrated, its use was probably beneficial and almost certainly not harmful.

D. Fabrication

1. Procedure

The fabrication sequence used for Kovar endcap lamps was essentially identical to that used for nickel endcap lamps, described in Section IV-D, with the additional nickel plating step on Kovar lamps performed prior to bake and fill operations on "latest technology" versions.

2. Yield

Six of eight fabrication starts on prototype lamps produced operable lamps. Four of seven starts on interim version lamps were successful. The in-process losses in these groups were generally due to leaks in brazed endcap seals across unwetted regions on the sapphire envelope seal surface. Yield on "latest technology" Kovar endcap lamps was five of five.

A summary of Kovar lamp fabrication activity is given in Table 7.

TABLE 7

SUMMARY OF KOVAR ENDCAP LAMP FABRICATION

Start No.	Serial No.	Design Type	K-Rb Ratio	Xenon Pressure (torr)	Disposition
20	476	Prototype	50:50	760	Life tests at ILC
21	477	Prototype	50:50	760	Failed in first operation
22	478	Prototype	50:50	760	Life tests at ILC
23	479	Prototype	50:50	760	Life tests at AFAL/405B
28	483	Prototype	100:0	760	Life tests at AFAL/405B
29	484	Prototype	90:10	760	Life tests at AFAL/405B
30	485	Prototype	90:10	760	Life test in vacuum at ILC
31	---	Prototype	-----	---	Leak in processing
32	486	Interim	10:90	760	Leak in processing
33	487	Interim	50:50	760	Life tests by Air Force
34	---	Interim	-----	---	Leak in processing
35	488	Interim	0:100	760	Life tests at AFAL/405B
36	---	Interim	-----	---	Seal leak after brazing; scrapped
37	490	Interim	50:50	1000	Laser tests
38	489	Interim	75:25	760	Life tests by Air Force
46	496	Latest Tech.	75:25	760	Life tests at ILC
47	497	Latest Tech.	75:25	760	Life tests at ILC
48	498	Latest Tech.	75:25	760	Life tests at ILC
49	499	Latest Tech.	75:25	760	Life tests at ILC
50	500	Latest Tech.	75:25	760	Laser tests, life tests by Air Force

E. Testing

1. Prototype and Interim Lamps

Of particular interest was on-off cycle durability of early Kovar endcap lamps since this lamp design type was inspired primarily by the need for better lamp cycle life than was, at that time, exhibited by nickel endcap lamps. Consequently, early life testing accented on-off cycling with run cycles of relatively short duration. As is shown in Table 8, the cycle lifetimes of prototype Kovar lamps, Numbers 476 and 478, were quite encouraging. Failures were unrelated to the brazed endcap seals. Subsequent tests on prototype lamps demonstrated the potential for extended lifetime in air. Lamp Number 484, for example, was

operated for 1000 hours with very few cycles. In general, the test results on prototype lamps indicated design deficiencies (discussed previously) in the endcap weld joints and electrode subassembly weld caps.

TABLE 8

LIFE TEST RESULTS FOR PROTOTYPE AND INTERIM DESIGN KOVAR ENDCAP LAMPS

Lamp No.	Lifetime Cycles	Lifetime Hours	Failure Mode
PROTOTYPE LAMPS			
476	85	158	Unlocated leak, probably in weld cap
478	183	287	Leak in endcap weld joint
479	20	300	Leak in endcap weld joint
483	25	145	Leak in endcap weld joint
484	4	1000	Fracture in sapphire adjacent to seal
485	4	60	Leak in weld cap
INTERIM LAMPS			
487	283	2012	Lamp still good; tests temporarily interrupted
488	81	852	Leak in endcap near seal at cathode end
489	240	1636	Envelope fracture along basal plane
490	---	----	Lamp not yet tested

The weld joint modification incorporated in interim version Kovar lamps substantially improved lifetime. Lamp Number 489 survived 1636 hours and 240 on-off cycles before the envelope failed due to basal plane cleavage cracking. Another interim version lamp tested, Number 488, failed due to a leak in the braze-affected region of the cathode endcap. The leak was probably induced by a combination of excessive erosion of the base metal by the brazing melt, finite cyclic stresses in that region, and oxidation. Most noteworthy are life test results on lamp Number 487, which has survived 283 cycles and 2012 hours and remains intact. This lifetime was the best achieved on the program.

2. Latest Technology Lamps

Life test results for "latest technology" Kovar endcap lamps are given in Table 9. Envelope failure by basal plane cleavage cracking occurred relatively early in the lifetimes of the two lamps from the first brazing lot, Numbers 496 and 497. Although pronounced cleavage cracks were present in the envelope of lamp Number 493 from the second "latest

technology" Kovar lamp lot, failure by leakage occurred in the endcap near the brazed seal much like in lamp Number 488 described earlier. The same endcap leakage failure occurred in lamp Number 498 from the second lot but, interestingly, no cleavage cracks were present in the envelope of this lamp. Instead, a whitish, frosty region developed over the anode half of the envelope at approximately the 500-hour point in lifetime when the auxiliary heater failed causing loss of K-Rb pressure and attendant operation for several hours in the "xenon arc mode" (see Section VI-B).

In general, the life test results on "latest technology" Kovar lamps indicate much the same kinds of life-limiting problems as were found in "latest technology" nickel endcap lamps; i.e., basal plane cleavage cracking of the sapphire envelopes and development of leaks in the braze-affected zones of the endcaps.

TABLE 9

LIFE TEST RESULTS FOR
LATEST TECHNOLOGY KOVAR ENDCAP LAMPS

Lamp No.	Lot No.	Lifetime Cycles	Lifetime Hours	Failure Mode
496	1	64	643	Envelope cracking along basal plane
497	1	59	475	Envelope cracking along basal plane
498	2	62	553	Leak in braze-affected zone of cathode endcap
499	2	85	853	Leak in braze-affected zone of cathode endcap
500	2	--	---	Not yet tested

Average Lifetime for Lot 1: 62 cycles/559 hours
Average Lifetime for Lot 2: 74 cycles/703 hours
Average Lifetime for Overall Group: 68 cycles/631 hours

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VI. GENERAL TOPICS

In the previous sections, the iterative development of PES, nickel endcap, and Kovar endcap lamps was described. These activities were devoted almost entirely to the improvement of the long term structural durability of the lamps. In this section, certain general topics common to all K-Rb laser pump lamps are discussed. Each topic represents an area for technical investigation that must be addressed in future work.

A. Sapphire Envelope Degradation

1. General Remarks

Since the commencement of work on the EFM-phase K-Rb lamp development, only cored, polished sapphire has been used for lamp envelopes. The material is obtained from Union Carbide, Crystal Products Department, in the form of solid rods, core drilled from boules grown by the Czochralski technique. Quality requirements imposed on the supplier, as reflected in an ILC procurement specification (PS 1006), dictate that boules of UV grade sapphire (a Union Carbide designation) be used that are specially selected on the basis of careful visual inspection to ensure the absence of inclusions, fine bubbles, and the like. All sapphire procured to date has been so-called 60^{o} material; i.e., with the crystallographic C-axis oriented at approximately 60^{o} from the rod (and boule) axis.

Machining of the rods to final envelope configurations is performed by Insaco, Inc. of Quakertown, Pennsylvania. The envelope bore is formed by core drilling, reaming, and fine polishing in successive steps. The external surface is then ground and polished to desired shape and finish. All machining is performed using diamond impregnated tools or diamond paste.

The internal wall of the tube is relatively difficult to polish; many fine scratches and occasional small gouges or heavier scratches remain after the polishing operation. It was previously believed (and may still be true) that these residual surface defects act as preferred sites for nucleation of aluminate crystallites (i.e., frosting). This belief prompted the development of a high temperature, hydrogen gas polishing technique on an earlier program at ILC[3]. Hydrogen gas polishing was effective in smoothing residual surface defects in the machined sapphire. Gas polishing work was discontinued when it was found that envelope frosting, at least early in lamp life, could be prevented by improved bake and fill methods and the use of uranium as a getter (which eliminated "free"

oxygen from the lamps). None of the envelopes on the present program were gas polished.

In some recent lamps, envelope frosting has been observed that seems to be promoted by higher than normal lamp operating temperatures. Such frosting occurs despite the use of a getter and improved lamp processing. In addition, basal plane cleavage cracking, a phenomenon possibly related to frosting, has become a primary failure mechanism in K-Rb lamps. These two subjects are discussed in detail below.

2. <u>Envelope Frosting</u>

It was previously suggested[4] that a chemical reaction can occur between potassium vapor and the sapphire envelope that is not dependent on the presence of "free" oxygen (e.g., O_2, H_2O, CO, etc.) in the lamp. Specifically the reaction

$$6K\ (g) + 34\ Al_2O_3\ (c) \longrightarrow 3\ \left[K_2O\text{-}11\ Al_2O_3\right]\ (c) + 2\ Al\ (1)$$

to form potassium beta alumina was proposed. A rough estimate of the free energy change for the above reaction as a function of temperature was made based on an extrapolation of published free energy data for the analogous sodium beta alumina compound, $Na_2O\cdot 11\ Al_2O_3$.

Recently, one free energy datum specifically for potassium beta alumina was found in the literature.[9] This datum indicated that potassium beta alumina was slightly more stable than sodium beta alumina, at least at 350°C. Free energy of reaction curves for the proposed potassium attack reaction have been carefully recalculated and are shown in Figure 14.

Because the free energy data for potassium beta alumina used to generate the curves were sparse and, at best only approximate, the reader is cautioned not to interpret the curves too literally. They are presented primarily to show that at least one potassium vapor attack reaction is plausible, thereby providing an explanation for experimentally observed "frosting" described below. The reader is also reminded that the degree of energetic favorability indicated by the reaction free energy curves (i.e., the magnitude of negative free energy values) is not directly related to reaction kinetics. Thus, although the reaction may be very energetically favorable at low temperatures and only marginally favorable at elevated temperatures typical of envelope operation, the reaction would more likely occur under the latter conditions where the high temperatures facilitate mechanisms such as diffusion, structual rearrangement, and the like.

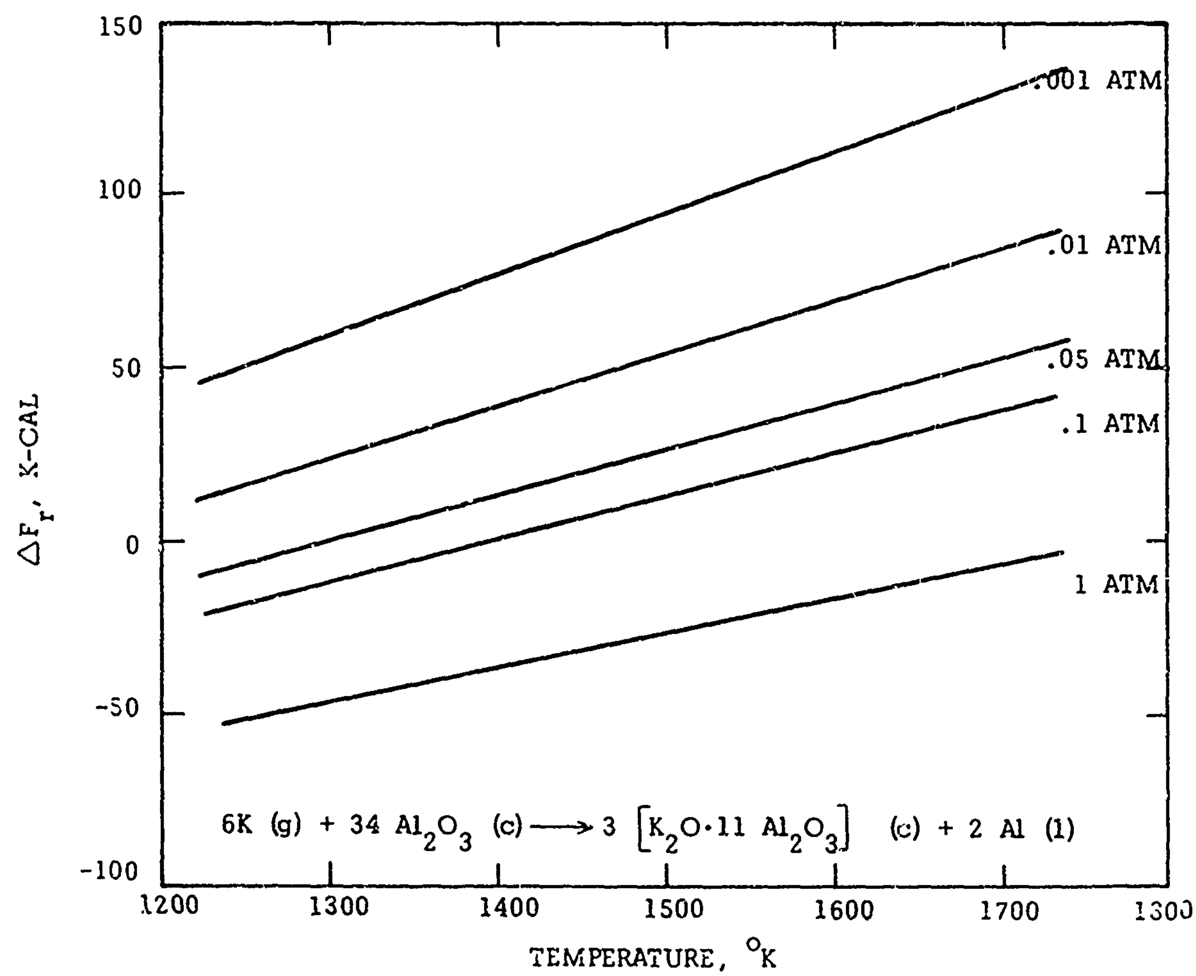

FIGURE 14. Estimated Reaction Free Energy for Potassium Beta Alumina Formation

In practice, reactions between potassium and sapphire result in frosting; i.e., the formation of aluminate crystallites on the lamp envelope interior surface. In most of the lamps tested on this program, visually discernible frosting did not occur. However, there are some experimental data that indicate that when lamp envelope temperature is increased by operating lamps in a vacuum rather than in ambient air, frosting occurs in relatively short times. Lamp Number 451, operated in a fused quartz vacuum jacket on the previous program, developed frosting in the form of distinct, separate crystallites which formed on the interior wall of the envelope well within 100 hours of operation. On this program, a very similar effect was observed on lamp Number 485 which was operated in an evacuated bell jar. This lamp failed after approximately 60 hours of operation due to loss of K-Rb through an endcap leak and subsequent electrode overheating. Scanning electron microscopy (SEM) of the sapphire envelope revealed the presence of many isolated crystallites that had grown on, and perhaps into, the sapphire suface as shown in Figure 15. Concurrent x-ray probe analysis (EDAX) showed the crystallites to contain potassium and aluminum as principal constituents (as had earlier analysis of crystallites on the envelope of lamp Number 451). In each instance of frosting, the crystallites that formed on the sapphire surface were for the most part aligned with the sapphire basal plane. Significantly, beta alumina structures are contiguous with alpha alumina structures in the basal plane directions. The crystallites were also hexagonal in shape. Beta aluminas have hexagonal crystal structures.

Another apparent manifestation of excessive envelope temperature was observed in lamp Number 498. In this instance, an extensive region of the envelope bore near the anode became covered with a continuous hazy coating during a time period of a few hours midway through life testing when the auxiliary heater failed causing the lamp to operate in a xenon dominated arc mode near the anode. SEM and EDAX analysis showed a severely "chewed up" sapphire surface (Figure 16) with no indication of potassium present. Conceivably, the high temperatures associated with the xenon arc caused surface melting or excessive surface transport on the sapphire.

In the ultimate satellite application, lamps must operate without benefit of free convection cooling due to the absence of gravity. In consequence, they will operate with higher envelope temperatures, other things being equal, than in standard life tests conducted in ambient air. Lamp testing in vacuum effectively simulates this condition; i.e., no convective cooling. As noted above, experience to date with lamps

FIGURE 15. SEM Photomicrograph of "Frosting" Crystallites (1000X)

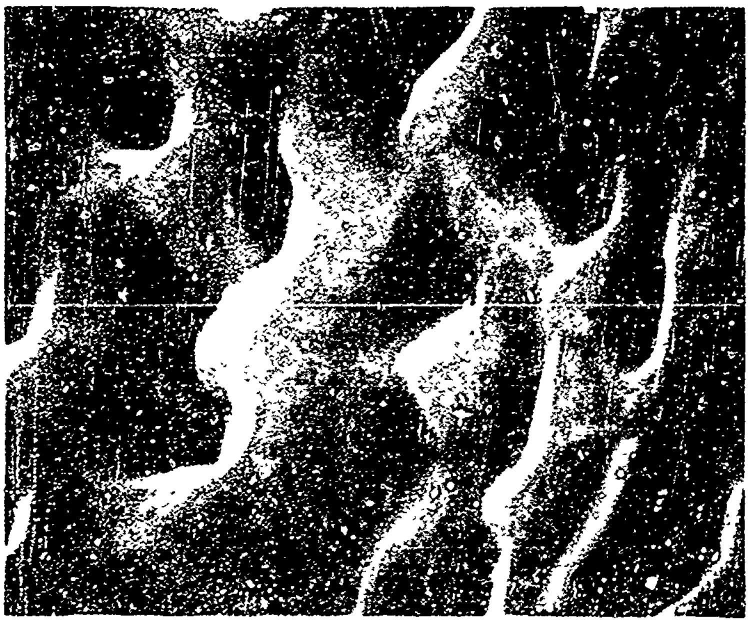

FIGURE 16. SEM Photomicrograph of Sapphire Envelope Interior in Region Frosted by Xenon Arc Mode (9000X)

operated in vacuum strongly indicates a tendency for envelope frosting to occur early in life. Additional lamp testing in vacuum must be conducted to confirm this.

If the frosting phenomenon proves to be a genuine problem at realistic envelope temperatures, various means of preventing its occurrence must be explored. These might include reduction in the potassium content of the fill, improvement in sapphire surface finish by gas polishing, a modification of the lamp thermal design to keep envelope temperatures within an acceptable range, or a change in crystallographic orientation of the sapphire. Basic studies of high temperature chemistry in the potassium-alumina system are also recommended in future work.

3. Cleavage Cracking

Until the final life testing of nickel and Kovar endcap lamps on this program, basal plane cleavage cracking in sapphire envelopes was more a nuisance than a serious problem. The occurrence of such cracking in nearly every recent life test lamp and the failure of several of the lamps as a result of such cracking now suggests that the phenomenon be given considerable attention.

Basal plane cleavage cracking was first observed in an earlier program on envelope assemblies after a high temperature brazing operation. The cracking was then attributed to defective sapphire. However, scanning electron microscopy of a cracked region in a brazed envelope assembly fabricated more recently showed that crystallites containing calcium and aluminum, which were largely oriented along the sapphire basal plane, had formed on the cracked sapphire surface. It was hypothesized that free calcium had been generated by a reducing action of the zirconium brazing alloy on the calcia-containing high alumina alignment rod used to hold the envelope assembly during brazing and had attacked the adjacent sapphire envelope to form calcium aluminate. The basal plane orientation of the cracks in the sapphire and of the second phase surface crystallites suggested that calcium beta alumina, $CaO \cdot 6\ Al_2O_3$, had formed. (As noted earlier, beta aluminas have crystal structures contingucus with alpha alumina along the close packed basal plane.) A mechanism involving growth of the beta alumina into the sapphire along basal planes and consequent stressing of the sapphire lattice to cause cleaving was proposed to explain the cracking effect.

For some time, lamps have been brazed using fixtures made from molybdenum or sapphire. Significantly, cleavage cracking of envelopes during brazing has not reoccurred.

As noted above, basal plane cleavage cracking has recently developed in lamp envelopes during life testing. As the average lamp lifetime has increased and especially during testing of "latest technology" lamps, cleavage cracking has become the rule rather than the exception. Nine lamps of ten that were recently operated beyond the 400 hour point had cleavage cracks. Perhaps, cleavage cracking in operating lamps can be explained by the same mechanism as was postulated earlier to explain cracking in brazed envelopes. In operating lamps, it is conceivable that potassium beta alumina is formed as microscopic nuclei that wedge into the sapphire basal planes. The plausibility of forming potassium beta alumina as the product of potassium attack on the sapphire was discussed in the section on envelope frosting.

Thus, cleavage cracking and envelope frosting may be two manifestations of the same basic chemical reaction. Perhaps frosting is the gross effect, occurring when the envelope temperature is sufficiently high to make general surface attack favorable. Cleavage cracking occurs when reaction kinetics are marginal for rapid growth of beta alumina crystallites on the sapphire surface and instead favor local nucleation and intrusion of the compound into the sapphire lattice.

Postmortem examination of envelopes from failed lamps using SEM and EDAX did not provide confirmation of the above hypothesis. Most cleavage cracks, when viewed on the inside surface of the envelope, appeared as open crevasses (Figure 17) with no signs of foreign crystallite intrusions. Incipient cleavage cracks were occasionally found that contained possible second phase "wedges" (Figure 18); however, it is equally possible that the foreign material had accumulated in the cracks after their nucleation. Certain lamp envelopes actually parted along cleavage planes providing exposed fracture faces for examination. These basal plane faces (Figure 19) had both rough and smooth areas, second phase (apparently aluminate) crystallites, and relatively high levels of elemental contaminants (including at various places or generally, Fe, Ni, Cr, Ca, Si, and Mg). These contaminants were also found on the envelope interior wall. Whether their presence is related to the cracking phenomenon is not known.

Other mechanisms that might cause basal plane cleavage cracking in sapphire must be considered as well. Unfortunately, such cracking is virtually unprecedented. There is no record in the literature of sapphire fracture on its basal plane, even when specific attempts are made to nucleate and propagate such cracks. It is possible that a thermal stress field peculiar to the K-Rb lamp geometry and temperature distribution induces slip (i.e., dislocation motion) on the basal plane in a

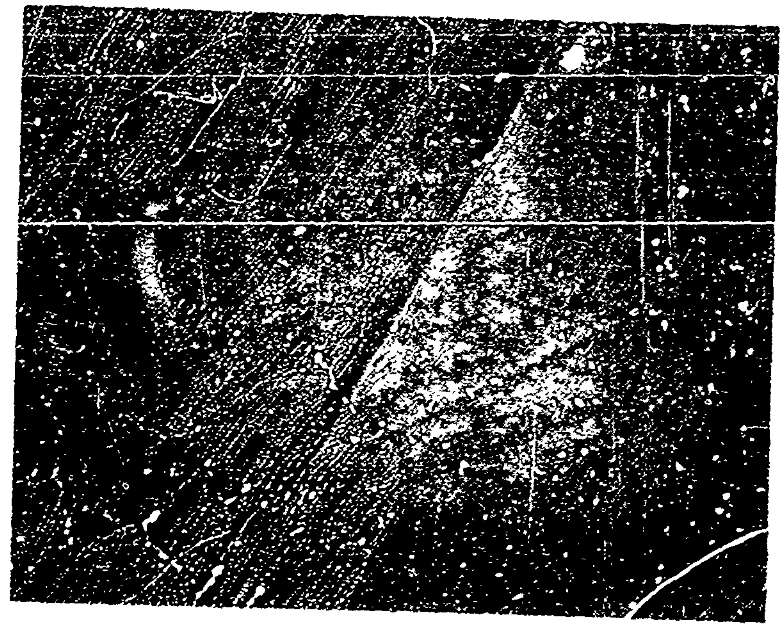

FIGURE 17. SEM Photomicrograph of Typical Basal Plane Cleavage Crack on Envelope Interior Surface (1400X)

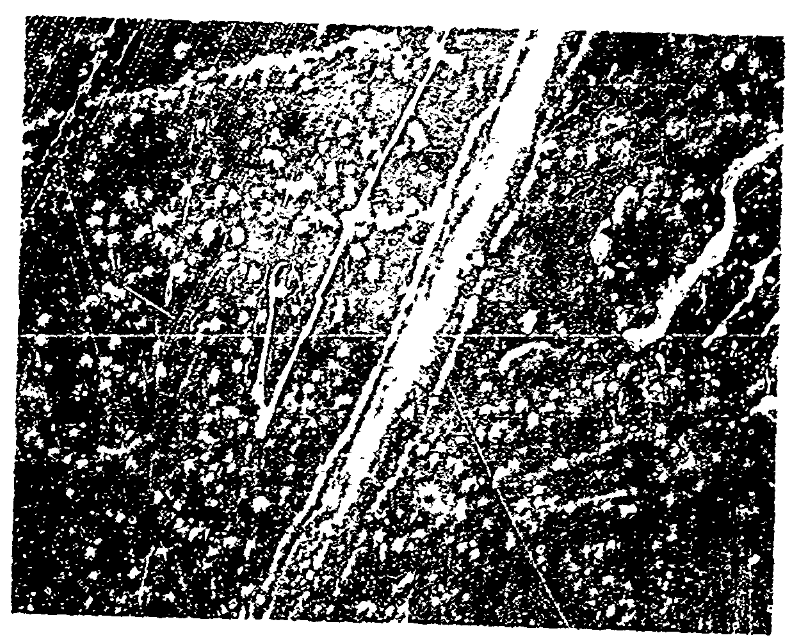

FIGURE 18. SEM Photomicrograph of Cleavage Crack with Possible Second Phase "Wedge" (10,000X)

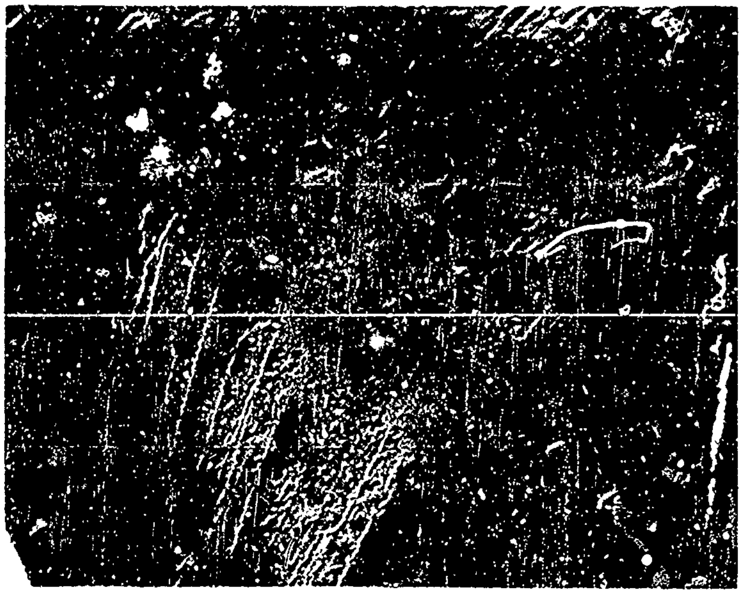

FIGURE 19. View of Basal Plane Cleavage Crack Face (125X) (Inside wall of envelope can be seen in the left hand corner of the picture.)

fashion that causes cracks to nucleate and grow, perhaps with help from the corrosive alkali metal environment. Experiments to separate the chemical and the thermo-mechanical environments imposed on the envelopes should be designed and conducted. In general, the envelope cleavage cracking problem requires primary attention in follow-on work.

B. Thermal Design

The two most important aspects of lamp thermal design are control of the peak envelope temperature and control of the K-Rb liquid reservoir temperature. The subject of thermal design has received little attention to date largely because other life-limiting problems were more visible, particularly intergranular leakage in endcaps, weldment cracking, and seal fractures.

As noted earlier, envelope temperature may be of critical importance in the rate of potassium attack. Experience to date indicates that as low a temperature as possible is desirable. However, general lamp design constraints such as input power, arc gap length, physical surroundings (i.e., reflective pump cavity, zero-g, etc.), and practical limitations on bore diameter and K:Rb fill ratios dictated by lamp efficiency requirements have the effect of restricting the thermal design exercise.

Some reduction in envelope temperature can possibly be achieved by increasing the outside diameter of the envelope. The effect of this is to increase the surface area from which envelope heat can be dissipated by radiation. As noted in the earlier section on nickel endcap lamp development, 4 mm bore lamps were fabricated with two outside diameters (5.5 mm and 6.5 mm) to test this scheme. Envelope temperature measurements on these two lamp types, and also on 5 mm bore lamps, are to be performed in the near future at McDonnell Douglas.

Another obvious means of reducing envelope temperature involves modifications to the laser pump cavity design rather than to the lamp, namely, forced convection cooling using a blower. This option may be unacceptable from an overall systems viewpoint.

The other and relatively independent aspect of lamp thermal design, reservoir temperature control, must also be addressed in future work. Ideally, the lamp must operate at the desired voltage and K-Rb pressure with a low input power to the auxiliary heater; e.g., 5 watts. This allows long term drifts up or down in reservoir temperature to be compensated for by nominal adjustments of heater power with minimal power drawn from the satellite electrical mains.

Control of reservoir temperature is complicated by the relatively strong influence of the surrounding environment. For example, lamps operated in reflective enclosures (e.g., laser pump cavities) have different heater power requirements to sustain a desired arc voltage than do the same lamps operated in a freely radiating environment. Air drafts and variations in free convection also have strong effects. In the long term, the operation of lamps in the zero-gravity environment of the satellite with attendant absence of the free convection component must also be accommodated.

These complications explain in part the substantial range of heater power levels required to maintain optimum voltage in recent life test lamps. Some "latest technology" lamps, for example, required 50 watts or more of heater power while others operated properly with 15-20 watts.

Low required heater power has the additional advantage that the effects of loss of heater power (e.g., due to burnout or the like) are minimized. The resulting slight drop in K-Rb pressure will not have a significant effect on pumping efficiency. Conversely, loss of heater power in a lamp requiring a large auxiliary heat input has catastrophic effects. K-Rb pressure drops substantially, causing pumping efficiency to fall off dramatically and, very often, the arc to become xenon-dominated with attendant electrode and envelope overheating.

An additional consideration in reservoir temperature control is responsiveness to heater power adjustments. In present lamps, a half-minute or more elapses before lamp arc voltage changes in response to a heater power adjustment. This slow response complicates the design of the lamp voltage-heater power feedback loop that will eventually be used in the flight hardware lamp power supply. Improved thermal contact between the heater and lamp proper (presently a slip fit) and perhaps a shorter thermal path between the heater and the reservoir should alleviate this problem.

C. Laser Pumping Efficiency

1. Previous Work

Prior to EFM-phase work, a fill of 50K-50Rb (by weight percent) and 100 torr of argon to facilitate starting was standard in ILC K-Rb lamps. With this fill, lamps with 1.9 inch arc gaps had acceptable pumping efficiency at 250 watts input.

In later work, the arc gap was increased to 2.5 inches to reduce thermal loading on the envelope. Some loss of laser pumping efficiency occurred because of the reduction in arc power per unit length and an attendant decrease in arc brightness. Subsequently, the efficiencies of 2.5 inch arc gap lamps were improved by substituting xenon for argon in the fill. Efficiency increased with increasing xenon fill pressure over the range from 100 to 760 torr. The beneficial effect of xenon is attributed to the lower thermal conductivity of the Xe-K-Rb mixture resulting from the much higher mass of xenon atoms. The lower thermal conductivity fill reduces heat dissipation from the arc by radial conduction to the envelope walls and increases radiated output from the arc column commensurately.

2. Effect of K:Rb Fill Ratio

On this program, a group of 5 mm bore lamps was filled with various ratios of potassium and rubidium plus 760 torr of xenon (the best xenon pressure as determined in previous work). The pumping efficiencies of these lamps were measured by AFAL/405B using the same procedure as had previously been used to evaluate the effects of xenon versus argon. Multimode output was measured from a standardized Nd:YAG laser. Lamps were tested sequentially in the laser. Two lamps in the test group had identical fills to provide a check on consistency of the results. One other lamp was tested twice to provide an additional check on the experimental consistency.

The results of these tests, summarized in Table 10, indicate quite clearly that pumping efficiency improves monotonically with increasing proportions of potassium in the fill (except for the pure rubidium case). The superiority of higher potassium fills is explained by their higher intensity in the "potassium" or shorter wavelength wing of the output spectrum in comparison with the 50-50 fill as shown in Figure 20. Outputs from high potassium and 50-50 fills are comparable at Nd:YAG excitation bands in the "rubidium" or long wavelength wing.

3. Effect of Higher Xenon Pressure

An experiment was conducted to determine whether the previously established trend of higher pumping efficiency with increasing xenon fill pressure continued beyond the previous 760 torr maximum. One lamp, Number 490, was filled with 50K-50Rb and 1000 torr xenon for a comparative efficiency measurement against lamp Number 487, filled with 50K-50Rb and 760 torr. Preliminary and inconclusive results with these lamps indicate that no gain (and perhaps a loss) in efficiency is obtained at 1000 torr.

TABLE 10

TEST RESULTS, LASER PUMPING EFFICIENCY VERSUS K-Rb RATIO

Lamp No.	K:Rb Fill Ratio	Optimum Voltage	Laser Power
483	100:0	69V	110 mW
483	100:0	69V	1150 mW (2nd Run)
484	90:10	67.5V	1115 mW
485	90:10	67V	1105 mW
489	75:25	68V	965 mW
491	50:50	67V	845 mW
487	50:50	67V	955 mW
482	25:75	68V	450 mW
482	25:75	70V	490 mW (2nd Run)
488	0:100	61V	790 mW

NOTE: All lamps filled with 760 torr xenon.

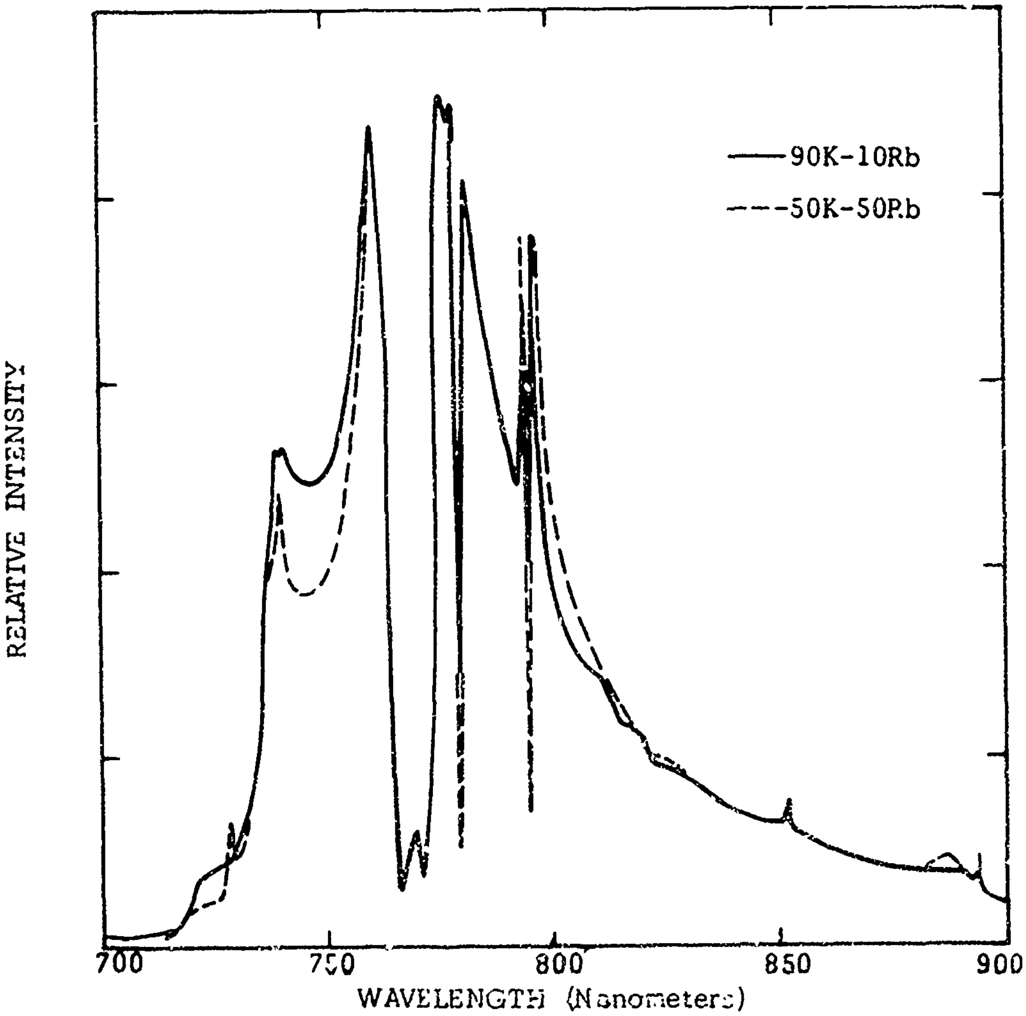

FIGURE 20. Comparison of High Potassium and 50K-50Rb Output Spectra

4. Effect of 4 mm Bore

Seven 4 mm bore lamps were fabricated late in the program as noted earlier. All were filled with 75K-25Rb and 760 torr xenon.

Laser pumping efficiency measurements were made using 4 x 5.5 mm, 4 x 6.5 mm, and 5 x 6.5 mm bore lamps having identical fills of 75K-25Rb plus 760 torr xenon (Numbers 500, 505, 510, respectively). These tests were carried out at GTE Sylvania, the laser subcontractor to McDonnell Douglas on the EFM program. Both multimode and TEM_{00} mode outputs from the laser were measured. As anticipated, the 4 mm bore lamps were significantly more efficient than the 5 mm bore lamps in these tests. The smaller envelope bore has the effect of constricting the wall stabilized arc somewhat which improves optical coupling into the laser rod.

Data from these tests are presently being reduced and are unavailable for inclusion in this report.

5. Discussion

The experimental findings described have important implications with respect to the development of long-lived (3000 hour) lamps. The use of high potassium fills is likely to aggrevate the problem of potassium attack of the envelope. As was shown in Figure 14, the free energy change of the attack reaction is very sensitive to potassium partial pressure. The higher envelope temperature that probably develops in the smaller bore 4 mm lamps will also tend to accelerate attack reactions Thus, it is conceivable that a trade-off between lamp efficiency and envelope life time will have to be made. Future work, both on the envelope attack problem and on lamp efficiency, will clarify this.

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